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## OBSERVATIONS ON THE PRECIPITATION OF MERCURY IN THE KJELDAHL METHOD

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In the determination of nitrogen by the Kjeldahl method one occasionally encounters a test in which the mercury precipitate in the distillation process is "off color" and the question immediately arises whether there was sufficient mercury precipitant added and how much this "off color" effect influenced the results of the test. When the mercury precipitating reagent is changed either for the sake of economy, for availability of materials, or for the purpose of avoiding sulphide fumes the question of the proper amounts of certain reagents to use in order to insure adequate precipitation of the mercury also arises. It is also urgent that something be known about the permissible limits of variation in the use of the prescribed reagents in order that the accuracy of the nitrogen results are not sacrificed. Finally, it would be decidedly helpful to the operator of the test if he could interpret the color of the mercury precipitable in terms of reliability of tests.

It may interest the cereal chemist to know that the original Kjeldahl method did not specify the use of mercury and that it was not until 1885 that Wilfarth suggested the use of mercury as an accelerator or catalyst for the Kjeldahl method. According to Fresenius (1904) the Wilfarth modification called for the use of 0.7 gram of HgO with the sulphuric acid. In order to precipitate the mercury as a sulphide the use of a quantity of potassium sulphide solution (40 grams per liter) in quantity greater than the corresponding amount of mercury used, was recommended. The sulphide solution was added to the digest after dilution. The Förster modification of the Kjeldahl method (Olson 1919) specified the use of 1 gram of metallic mercury. To precipitate the mercury the digest was diluted, and to it was added 25 cc. of a 40-gram-to-the-liter solution of commercial sodium sulphide.

In other text books on chemical methods which describe the Kjel-

dahl method one finds different recommendations with respect to the amounts of mercury to use as a catalyst as well as variable recommendations with respect to the precipitating procedure. Treadwell-Hall (1907) recommend the use of a few drops of metallic mercury. To the diluted digest is added 80 cc. of caustic soda solution and sufficient potassium sulphide (40 grams of salt per liter) to completely precipitate the mercury and cause the solution in the flask to appear black. About 25 cc. of potassium sulphide solution is recommended. Leach (1920) suggests the use of 0.7 gram of mercuric oxide or an equivalent amount of metallic mercury. The mercury is precipitated with 25 cc. of a potassium sulphide solution (40 grams of the commercial salt in a liter of water). The sulphide is added to the diluted digest. Scott (1925) advises the use of 0.7 gram of metallic mercury. The diluted digest is strongly alkalinized by the addition of sodium hydroxide—sodium sulphide solution (about 75 cc. of a mixture containing 25 grams of NaOH and 1 gram of  $\text{Na}_2\text{S}$ ). Griffin (1927) specifies the use of 0.7 gram of  $\text{HgO}$  or its equivalent in metallic mercury. The mercury is precipitated by the use of 50 cc. of potassium sulphide solution (40 grams to the liter) or 50 cc. of sodium thiosulphate solution (80 grams to the liter). In the book of Methods of Analysis of the Association of Official Agricultural Chemists (1925) it is specified that 0.7 gram of mercuric oxide or its equivalent in metallic mercury be used in each test by the Kjeldahl method. The mercury is precipitated in the diluted digest by adding 25 cc. of a potassium or sodium sulphide solution (40 grams commercial potassium or sodium sulphide per liter of water) or 25 cc. sodium thiosulphate solution (80 grams per liter). In the Kjeldahl-Gunning-Arnold method 0.7 gram of mercuric oxide, or its equivalent in metallic mercury is used. In the last named method the mercury is precipitated by the use of double the amount of same strength solution as just mentioned for the official Kjeldahl method.

The Kjeldahl method as described in the Book of Methods of the American Association of Cereal Chemists calls for the use of 0.5 gram of red mercuric oxide as a catalyst. Precipitation of the mercury is accomplished by the use of sodium or potassium sulphides, or sodium thiosulphate of the usual strength. The precipitating reagents are mixed with the alkali and withdrawn with it.

Paul and Berry (1921) in their studies with the Kjeldahl method recommended the use of 20 cc. of a 4% solution of sodium or potassium sulphide for the precipitation of a 0.5 to 0.7 gram of mercury.

Coleman, Dixon, and Fellows (1925) found potassium and sodium sulphide in general use as mercury precipitants in the protein testing of cereal products. They stated that the advantage of sodium thio-

sulphate as a mercury precipitating agent lies in the absence of sulphide odors and that 1 gram of the salt is sufficient to precipitate 0.9 gram of metallic mercury.

### Material Used in This Investigation

The investigation recorded in this paper was made relative to the use of two amounts of mercury as a catalyst, namely, 0.5 gram and 1 gram of metallic mercury. The material used for digestion was a hard wheat clear grade flour, a soft wheat flour, and 0.1 gram of ammonium sulphate. The salts used for precipitating reagents were C. P. crystalline sodium sulphide ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ), C. P. crystalline sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ), potassium sulphide (liver of sulphur) U. S. P. X., and sodium sulphide 60%, fused chips. The sulphide salt, either potassium or sodium, was made up in the concentration of 40 grams per liter, while the sodium thiosulphate salt was made up in the concentration of 80 grams per liter.

The precipitation of the mercury in each case was accomplished by adding an aliquot portion of each of these solutions to the sodium hydroxide solution which had been previously prepared so as to bring the digest well on the alkaline side. The other conditions of the test were those specified in the Book of Methods of the A. A. C. C.

### Amount of Ammonia Held Back When No Precipitating Reagent is Used

The sample of hard wheat clear flour, soft wheat flour, and 0.1 gram of ammonium sulphate were digested in the usual manner with 0.5 gram and 1 gram of metallic mercury respectively. Distillation was accomplished without the use of mercury precipitants in order to learn how much ammonia was held back and did not distill over. In Table I

TABLE I  
THE AMOUNT OF AMMONIA HELD BACK WITH NO MERCURY PRECIPITATING AGENT  
USED IN THE KJELDAHL METHOD

Number of tests	Sample of	Weight mercury	Average protein no pcpt. agent $\text{N} \times 5.7$	Protein official method $\text{N} \times 5.7$	Ammonia held back
		grams	P.ct.	P.ct.	P.ct.
3	Hard wheat clear flour	.5	11.20		14.9
3		1.0	12.62		4.1
12		.5		13.16	
4	Soft wheat flour	.5	7.32		13.0
4		1.0	7.90		5.9
2		.5		8.40	
5	0.1 gm. ammonium sulphate and 1.25 gram sugar	.5	9.60		20.0
5		1.0	11.36		5.4
2		.5		12.00	

it will be found that when 0.5 gram of metallic mercury was used 14.9%, 13%, and 20% respectively, of the ammonia was held back and did not distill over. These percentages correspond with those of Paul and Berry (1921) who found that from 12% to 15% of ammonia was held back when no precipitating agent was used with 0.7 gram of mercury, and also with the findings of Coleman, Fellows, and Dixon (1925), who noted that 13% of the ammonia was held back with no mercury precipitating agent used. When the amount of mercury was increased to 1 gram, the results were somewhat different than one would expect, in that the larger amount of mercury held back a lower percentage of the ammonia. Compared to the former figures of 14.9%, 13%, and 20%, a retention of 4.1%, 5.9%, and 5.4% respectively was observed. This change in percentage of ammonia held back with an increase in concentration of mercury is possibly caused by a larger percentage of mercuric over mercurous compounds present in the digest of greater concentration and stronger oxidation.

#### **Effect of Increasing Amounts of Precipitating Reagent on Protein Test Results**

An investigation was made of the amount of precipitating agent actually necessary to give maximum protein results with the two amounts of mercury used in the above tests. It was thought that this precipitating reaction, which is generally considered to be quantitative, might have some colloidal characteristics which would alter the theoretical data and have an affect on the nitrogen results. Also the variations in the color of the mercury precipitate which were obtained in the boiling solution during distillation were studied for any relationship they might have to the proper use of the precipitant.

#### **Results with Increasing Amounts of Sodium Sulphide**

In these tests the sample of hard wheat clear flour and 0.125 gram of ammonium sulphate plus 1.25 grams sugar were used as sources of nitrogen. In Table II, and in Figure 1, it will be noted that when 0.5 gram of mercury was used and the amount of sodium sulphide increased, the first result which compares favorably with the maximum protein result is obtained from the use of 0.6 gram of sodium sulphide per test, or 15 cc. of the solution of sodium sulphide precipitant. When the amount of mercury catalyst was raised to 1 gram, the amount of sodium sulphide necessary for maximum results was increased to practically 1.2 grams or 30 cc. of the solution of sodium sulphide precipitant. These results check very closely with the theoretical reaction in which the amount of precipitant necessary to complete the precipi-



tation of 1 gram of mercury is 1.1978 grams, and for 0.5 gram of mercury is 0.5989 gram.

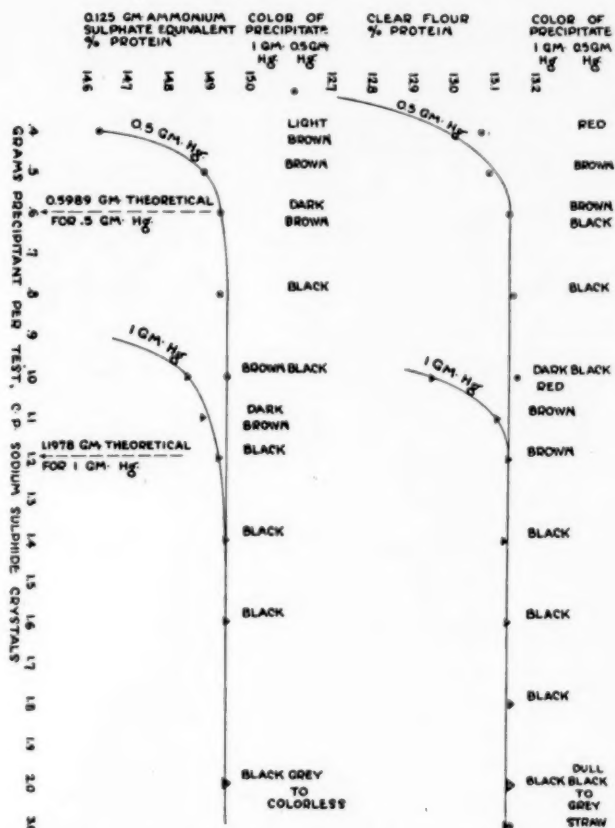


Fig. 1. The amount of C. P. sodium sulphide crystals (Na<sub>2</sub>S.9H<sub>2</sub>O) necessary to precipitate mercury in the Kjeldahl determination.

In this series of tests it was observed that maximum protein results could be obtained when the color of the precipitate was not distinctly black, although it appears as if a black precipitate is good insurance that sufficient sulphide has been added. When using 0.5 gram of mercury, and increasing the sodium sulphide per test from 2 to 3 grams, the precipitate tended to range in color from a grey to a bright straw color. The protein results were normal and no excessive sulphide fumes were noticeable in the receiving flasks. The color of the precipitate, however, was conclusive of the fact that an excess of sulphide had been used.

TABLE II

THE AMOUNT OF C. P. SODIUM SULPHIDE CRYSTALS NECESSARY TO PRECIPITATE MERCURY IN THE KJELDAHL DETERMINATION

Grams of precipitant per test	Using 0.5 gram Mercury			Using 1.0 gram Mercury		
	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$
Clear Flour						
.2	3	light red	12.15			
.3	3	red	12.62			
.4	6	red	13.08			
.5	3	brown	13.10			
.6	6	brown-black	13.15			
.8	6	black	13.16	3	red	11.26
.9				3	dark red	11.83
1.0	6	black	13.17	3	dark red	12.96
1.1				3	brown	13.12
1.2				6	brown	13.15
1.4				3	black	13.14
1.6				6	black	13.15
1.8				3	black	13.16
2.0	9	dl. black to grey	13.16			
3.0	3	bright straw	13.16	6	black	13.15
0.125 gram Ammonium Sulphate						
.3	3	light brown	14.20			
.4	3	light brown	14.64			
.5	3	brown	14.90			
.6	3	dark brown	14.94			
.8	3	black	14.94	3	brown	13.92
1.0	3	black	14.96	3	brown	14.86
1.1				6	dark brown	14.90
1.2				6	black	14.94
1.4				3	black	14.96
1.6				3	black	14.96
2.0	3	gray to colorless	14.96	3	black	14.96

The theoretical amount of precipitant necessary for 0.5 gram mercury equals 0.5989 gm. That for 1 gm. Hg. equals 1.1978 gms.

### Results with Increasing Amounts of Sodium Thiosulphate

In these tests the source of nitrogen was the same as in the above series of tests with sodium sulphide. In Table III, and from Figure 2, it will be observed that when 0.5 gram of mercury was used with increasing amounts of sodium thiosulphate, the first result which compared favorably with the maximum protein result was obtained by using approximately 1.4 grams of the sodium thiosulphate per test, or 17.5 cc. of solution. When the amount of mercury catalyst was increased to 1 gram, approximately 2.8 grams of sodium thiosulphate or 35 cc. of solution was necessary for maximum protein results. This



TABLE III

THE AMOUNT OF C. P. SODIUM THIOSULPHATE CRYSTALS NECESSARY TO PRECIPITATE MERCURY IN THE KJELDAHL DETERMINATION. PRECIPITANT ADDED WITH THE ALKALI

Grams of precipitant per test	Using 0.5 gram mercury			Using 1.0 gram mercury		
	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$
Clear Flour						
.8	3	light red	12.88			
1.2	3	red	13.08			
1.4	5	red	13.10			
1.6	3	dull red	13.13	3	light red	12.88
2.0	12	brown	13.13	3	red	12.98
2.4	6	brown-black	13.12	3	dull red	13.08
2.6	3	brown-black	13.14	3	dull red	13.10
2.8				5	brown	13.13
3.2	2	black	13.14	5	brown	13.12
3.6			13.14	5	brown-black	13.14
4.0	4	black	13.14	2	black	13.14
6.0	2	black	13.15	2	black	13.16
8.0	2	black	13.15			
0.125 grams Ammonium Sulphate						
.8	3	light red	14.68			
1.0	3	light red	14.72			
1.2	6	light red	14.87			
1.4	2	red	14.92			
1.6	6	dull red	14.94	3	dull red	14.16
2.0	3	dull red	14.92			
2.4				3	dull red	14.84
2.6				3	red	14.88
2.8	3	brown-black	14.94	5	red	14.94
3.2	3	black	14.96	6	red	14.94
3.6				3	dull brown	14.96
4.0	3	black	14.96	3	brown	14.96
8.0	2	black	14.94			

In solutions of mercuric salts and thiosulphates when the ratio of the concentration of the two salts is 1:1 the most normal reaction is for one mole of sodium thiosulphate to precipitate one mole of mercuric sulphide (black) and in such a reaction the theoretical requirement would be 1.237 grams of the sodium thiosulphate crystals to precipitate 1 gram of mercury. The actual requirements for adequate precipitation in the Kjeldahl method are somewhat greater than this theoretical requirement. However, it is possible that this is not a true picture of the reaction in these tests for as Allen and Crenshaw (1912) have shown the product of the action of soluble thiosulphates on mercuric salts depends upon the ratio of the two salts in the solution and also on their concentration. In the light of these results a smaller amount of the sodium thiosulphate precipitant is necessary for adequate precipitation when the precipitating reaction is carried out in the

dilute acid solution of the digest than in the condition that exists when the alkali and precipitant are mixed and added to the diluted digest.

From the nature of the curves, with increasing amounts of sodium thiosulphate, it will be observed that slightly low protein results follow the use of a slightly insufficient amount of precipitant. Failure to use large enough amounts of this precipitating agent has no doubt discouraged its use in many cases.

TABLE IV

THE AMOUNT OF POTASSIUM SULPHIDE, FUSED CHIPS, U. S. P. X. (LIVER OF SULPHUR) NECESSARY TO PRECIPITATE MERCURY IN THE KJELDAHL DETERMINATION

Grams of precipitant per test	Using 0.5 gram mercury			Using 1.0 gram mercury		
	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$	Number of tests	Color of mercury precipitate	Per cent Protein $N \times 5.7$
Clear Flour						
.2	3	red	12.82			
.3	3	dark brown	13.04			
.4	3	black	13.16			
.6	3	black	13.16	3	dark brown	12.90
.7	3	black	13.16	3	brown-black	13.02
.8				3	black	13.16
.9				3	black	13.14
1.0				3	black	13.16
1.2	3	black	13.16	3	black	13.16
1.6				3	black	13.18
2.0	3	grn.-black	13.16			
3.0	3	grn.-black		3	grn.-black	13.18
0.125 gram Ammonium Sulphate						
.2	3	red	14.74			
.3	3	dark brown	14.86			
.4	5	black	14.96			
.6	3	black	14.94	3	dark brown	14.74
.7				3	brown-black	14.82
.8	5	black	14.95	3	black	14.92
.9				3	black	14.92
1.0	3	black	14.98	3	black	14.94
1.2				3	black	14.96
1.6				3	black	14.98
2.0	3	grn.-black	14.98			
3.0	3	grn.-black	14.98	3	grn.-black	14.98

The color of the precipitate in the determinations with increasing amounts of sodium thiosulphate is less reliable as an index to whether the proper amount of precipitating agent was used. In the case of the clear flour, maximum results were obtained when the precipitate was dull red and brown. However, brown-black and black precipitates were the rule when sufficiently large amounts of sodium thiosulphate were used to insure adequate precipitation. In the case of increasing



the amounts of thiosulphate with the sample of ammonium sulphate and sugar, the brown-black and black precipitates were slower to appear. This is attributed to the fact that this sample did not produce oxidation reactions in the digestion equivalent to the oxidation of the flour sample, consequently the reaction when the precipitant is added, would not be brought to the same condition as found with the flour sample. When the precipitant is added to the diluted digest before the alkali is added, the brown-black and black precipitates indicating

TABLE V

THE AMOUNT OF C. P. SODIUM THIOSULPHATE CRYSTALS NECESSARY TO PRECIPITATE 1 GRAM OF MERCURY IN THE KJELDAHL METHOD

Grams of precipitant per test	Precipitant added with the sodium hydroxide solution			Precipitant added before the sodium hydroxide solution		
	Number of tests	Color of precipitate	Per cent Protein $N \times 5.7$	Number of tests	Color of precipitate	Per cent Protein $N \times 5.7$
Clear Flour						
1.4	3	light red	12.88	3	light red	12.94
1.6				3	red	13.04
1.8				3	red	13.08
2.0	3	red	12.98	3		13.14
2.2				3	dark red	13.14
2.4				3	dark red	13.14
2.6	3	dull red	13.10			
2.8	5	brown	13.13	3	brown	13.16
3.2	5	brown	13.12			
3.6	5	brown-black	13.14			
4.0	2	black	13.14			
6.0	2	black	13.16			
0.125 gram Ammonium Sulphate						
1.4	3	dull red	14.16	3	light red	14.72
1.6				3	red	14.84
1.8				3	dark red	14.92
2.0	3			3	brown	14.94
2.2				3	dark brown	14.96
2.4				3	dark brown	14.96
2.6	3	dull red	14.84			
2.8	3	red	14.88			
3.2	5	red	14.94	3	brown-black	14.96
3.6	6	red	14.94			
4.0	3	dull brown	14.96			

adequate precipitation, appear with smaller amounts of the sodium thiosulphate precipitant than when the alkali and precipitant solutions are mixed and added to the diluted digest. This indicates that more mercuric sulphide precipitate is formed and that with equal amounts of the sodium thiosulphate and equal amounts of mercury the reaction is more nearly completed in the former case.

When the amount of sodium thiosulphate was increased to 8 grams per test on these two samples, the precipitate was black and the protein results were normal. This amount was considered to be higher than any laboratory would use, but it indicates that large amounts have no depreciable effect on the reliability of result.

### Results with Increasing Amounts of Commercial Sulphide Salts

Potassium sulphide, fused, U. S. P. X. (liver of sulphur), was first used in these tests because the C. P. potassium salt was not available. Liver of sulphur is the product used in many laboratories and it was thought advisable to obtain figures on the use of this precipitant. In Table IV it will be observed that in using 0.5 gram of mercury the minimum requirement for potassium sulphide was 0.4 gram or 10 cc. of the solution per test to obtain maximum protein results. This requirement was 0.8 gram when the amount of mercury was raised to 1 gram.

When insufficient amounts of the precipitating agent were used the colors of the precipitate was red, brown, and brown-black. The best use of the precipitant gave a black precipitate while large amounts gave a precipitate of green-black color with no detrimental effects on the protein result.

Sodium sulphide, 60%, fused chips, when used in increasing amounts, gave figures equivalent to those obtained with potassium sulphide except that large amounts of the precipitant liberated sulphide fumes which, when passed into the receiving flask, deposited a colloidal precipitate of sulphur. This precipitate interfered with the best use of the indicator. This excess, while not disastrous to the protein result, is to be avoided. A black precipitate is an index to the proper use of this precipitant while a grey to colorless distilling solution is an index to the use of excessive amounts.

### Summary

1. In the Kjeldahl method, for maximum protein results, the minimum amount of precipitating agent for 1 gram of mercury was found to be approximately 1.2 grams of C. P. sodium sulphide crystals ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ) or practically the theoretical requirement and in the case of C. P. sodium thiosulphate crystals ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) approximately 2.8 grams when the precipitant is added with the alkali and approximately 2 grams when the precipitant is added before the alkali. The minimum amount of potassium sulphide, fused chips, U. S. P. X. (liver of sulphur) and sodium sulphide, 60%, fused chips, was approximately 0.8 gram for the precipitation of 1 gram of mercury.

2. The use of mercury precipitating agents in excess of the minimum requirement is good insurance of maximum protein results. In the case of sodium thiosulphate a generous excess is recommended while in the case of commercial sodium sulphide a slight excess appears to be most satisfactory.

3. In the regular Kjeldahl method the color of the precipitate during distillation is a fairly reliable index to the proper use of the precipitating agents as used in these tests. Black precipitates indicate adequate precipitation except in the case of sodium thiosulphate where a brown-black or black indicates the use of a sufficient amount.

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# ON THE SEPARATION OF "GASSING POWER" (DIASTATIC ACTIVITY) FROM "STRENGTH" IN BAKING TESTS

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## Introduction

In a paper published a year ago in this journal Fisher and Halton (1929, 1929a) pointed out *that the gassing power of a flour is independent of its strength and should be regarded as a separate problem from strength.* In the same paper Fisher and Halton further remark that this separation of gassing-power from strength "is perhaps the biggest positive advance that has yet been made in the study of strength" and they state how this advance was due to Wood (1907) and, particularly, to Humphries (1909), (1911), and (1920). On the whole, the notion popularly termed "strength of a flour" must be said to be a rather conglomerate one, and it is evident that the isolation of any of the constituents of this conglomerate for separate study is a very important thing in facilitating the understanding of the "strength" problem.

Studying Humphries' (1920) reports to the English Home-Grown Wheat Committee one cannot but admit that the separation of gassing-power from strength has been a big positive advance in the field of cereal chemistry.

The works of Humphries, and Wood have been cited and discussed frequently in the literature, Bailey (1925, p. 231) has discussed their researches in a very comprehensive way. In spite of the facts, however, that much publicity has been given to the work of Humphries and Wood, it may be said that Humphries' recommendation with regard to the importance of the separation of "gassing-power" from "strength" of a flour has not generally been followed. Recently the author tried out several of the baking-test-methods described in the literature. The author could not help being surprised at seeing, that many of these baking tests were of such a nature that the gassing-power of the flour was not separated at all from strength. On the contrary, it was found that the volume of the finished test-loaf was often the function of the gassing-power of the flour as well as of its strength.

In the present paper a control method, which was worked out by the author for deciding whether a given formula for making baking-tests is of such a nature that gassing-power is separated from strength, will be described. Furthermore, the results obtained by applying this control method to several baking-test-methods which are described in the literature will be given.

Before proceeding with the investigational phases of this paper, the author should like to make some remarks relative to the term: "gassing-power of a flour." This term is rather inadequate in as far as the flour itself by no means produces gas. It is the yeast which produces the gas and the role of the flour is simply (by means of its diastase) to supply the yeast with fermentable sugar from which the yeast may produce carbon dioxide. There can be no doubt, that the flour-properties, which Humphries terms "gassing-power" are the same as those covered by the word "diastatic activity," provided that "diastatic activity" is defined as the figures obtained, when using Rumsey's (1922) fundamental method or one of the modifications of this method. The author, therefore, is of the opinion that the term "gassing-power" may be substituted by the term "diastatic activity." In the present paper the two terms are used interchangeably, their meaning being the same.<sup>1</sup>

The following should be emphasized — *If the gassing-power (diastatic activity) is to be separated from strength in making baking-tests, there must not be a shortage of fermentable sugar, at any time during the dough-time. On the contrary, at every moment during dough-time there must be an excess of fermentable sugar present. The CO<sub>2</sub>-production of the yeast must not be limited at all by the amount of fermentable sugar present.* If this condition is fulfilled, then, and only then, will the volume of the finished test-loaf furnish a measure of the strength of the flour. If, on the contrary, the amount of CO<sub>2</sub> produced by the yeast is reduced owing to a shortage of fermentable sugar, in other words, if the yeast *starves*, the volume of the finished loaf will be a function of the diastatic activity as well as of the strength of the flour. Under such conditions a segregation of strength from gassing-power as laid down by Humphries has not been made, and the baking-test loses much of its value.

The question then arises, how is it possible to decide in practice by following a given formula for making baking-tests whether gassing-power has actually been separated from strength? The discussion in Part 1, of the present paper will deal with this question.

## Experimental

### PART 1

#### HOW TO DECIDE WHETHER A GIVEN FORMULA FOR MAKING BAKING-TESTS SEPARATES GASSING-POWER FROM STRENGTH.

When a dough is mixed, the diastatic enzymes of the flour immediately commence their work, i.e., produce maltose. The yeast on the contrary does not start its work quite so fast, as a matter of fact some

<sup>1</sup> It will be known that the figures obtained in using Rumsey's autolytic method represent the combined action of two sorts of enzymes: liquifying and saccharifying enzymes, Kent-Jones, (1927, p. 368). In all parts of the present paper the terms "diastatic" and "diastase" cover this combined action of the two sorts of enzymes.



time may elapse before the yeast is working at full speed. If the diastatic activity is of such magnitude that at every moment during dough-time there is produced as much maltose as the yeast is able to consume, shortage of fermentable sugar will, naturally, never occur. If, on the other hand the diastase in the flour does not produce as much maltose per unit of time as the yeast is capable of consuming, shortage of fermentable sugar may occur, but not necessarily so. This reservation is due to the fact that the dough contains a certain reserve-stock of fermentable sugar. This reserve-stock consists of: (1) The fermentable sugar existing in the flour before the dough was mixed—this is always a very small quantity; (2) the surplus of maltose produced in the dough at the beginning of the dough-time before the yeast is working at full speed; and, (3) the amount of fermentable sugar such as cane-sugar, which is intentionally added to the dough as an ingredient. However, it is well-known that the addition of such fermentable sugars to the dough is not practised in all countries. Shortage of fermentable sugar will or will not occur depending on whether or not this reserve-stock is sufficient. This in turn depends on the length of the dough-time, the amount of yeast, the dough-temperature, etc.

At all events it should be understood, that it is during the later stages of the dough-time, and especially during the proofing-period, that there may be danger of starvation of the yeast due to a shortage of fermentable sugar. If the proofing-period is finished without yeast starvation having taken place, one can hardly imagine that there will be danger of starvation occurring after the dough has been brought into the oven. In the oven the diastatic activity increases very rapidly and is not seriously interfered with before a temperature of about 63° C. has been reached, whereas the activity of the yeast falls off at a much lower temperature.

From what has just been said it will be understood that *in deciding whether a given baking-test-method is of such a nature that the gassing-power is kept separated from the strength, we may confine ourselves to examining whether the yeast starves during the proofing-period.*

The method that the author uses in deciding whether this is the case is given in the following section.

### Method of Study

The dough is made from its components in the proportions stated in the formula to be studied. Sufficient flour should be taken into operation, so that at least 850 grams of dough are obtained. The dough-fermentation and the punches, etc., are carried out exactly as stated, in the method under examination, up to the moment when the dough is to be moulded and brought to proof. Instead of being placed in the proofing cabinet the dough is handled in the following manner:

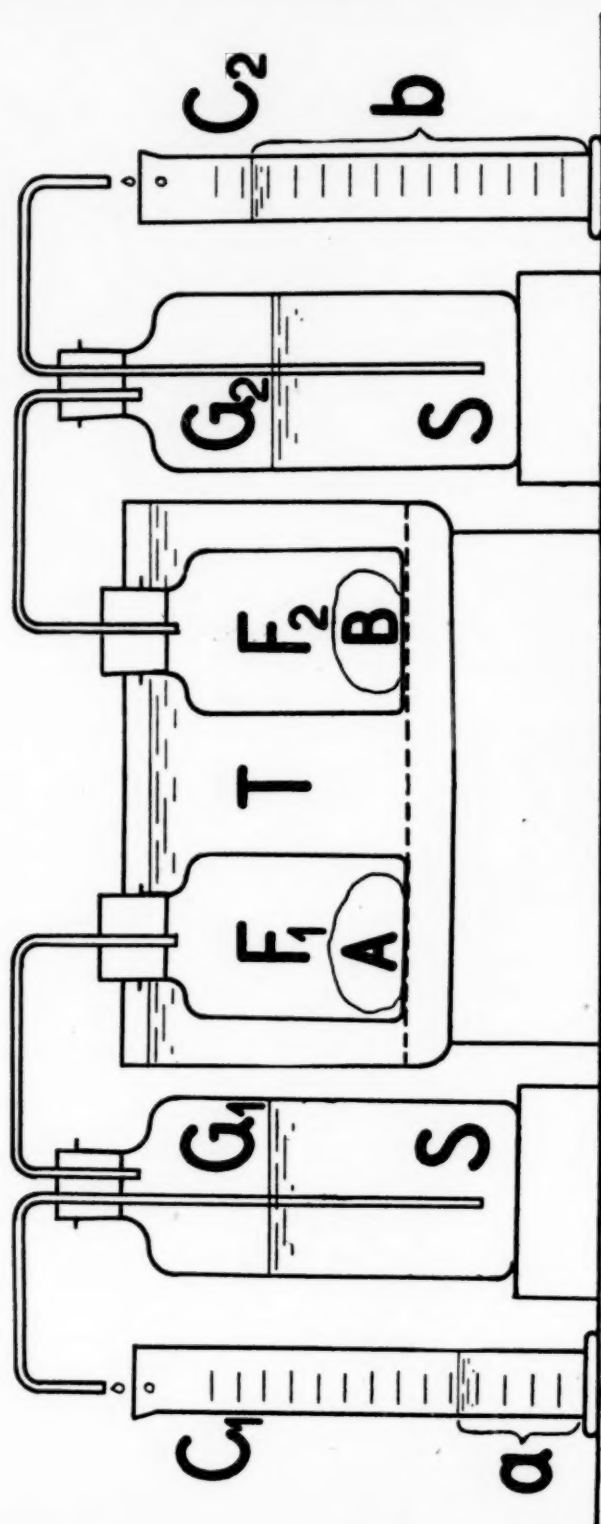


Fig. 1. Apparatus used in this study.

Two pieces, *A* and *B*, weighing 400 grams each are weighed off from the dough. 5 cc. of water is mixed into dough-piece *A*. This may be done by hand, or preferably in a small mixing machine. 5 cc. of a maltose solution (60 grams of pure maltose (hydrate) in 100 cc. of water) is mixed into dough-piece *B*. The 5 cc. of maltose solution corresponds to 3 grams of maltose. In carrying out these mixings care must be taken that the dough-pieces do not cool.

Use is next made of the well-known apparatus shown in Figure 1, which has been described by Jago (1911) and later on by Kent-Jones (1927). The piece of dough *A* is placed in the bottle *F*<sub>1</sub> and the piece of dough *B* in bottle *F*<sub>2</sub>. Should the dough-pieces become a little sticky on account of the addition of the 5 cc. of liquid, they may be dusted with a little pure wheat-starch, whereby they can easily be placed into the bottles.

The water-bath *T* is kept at the temperature at which the proofing would take place according to the formula under examination. One may say that the two dough-pieces are proofed in the bottles instead of in the proofing cabinet.

The amount of carbon dioxide evolved from either of the two dough-pieces is measured. The measurement of the carbon dioxide evolved is stopped at exactly the same moment as the proofing in the proofing cabinet would have been finished according to the formula which is under examination.

Let us assume, that dough-piece *A* has evolved *a* cc. of CO<sub>2</sub> and dough-piece *B* *b* cc. Now if *b* is greater than *a* the yeast in dough-piece *A* has evidently starved; and according to what was explained before this means that the formula used is of such a nature that the gassing-power (diastatic activity) is not separated from strength. The method described being a technical one, small differences between *a* and *b* up to approximately 25 cc., should not be regarded as significant.<sup>2</sup> Relatively large sizes of dough (400 grams) are chosen in order to suppress influences from different sources of errors.

As to the apparatus shown in Figure 1, either of bottles *F*<sub>1</sub> and *F*<sub>2</sub> will hold about 1 litre, and either of bottles *G*<sub>1</sub> and *G*<sub>2</sub> about 4 litres. The liquid *S*, which is displaced by the carbon dioxide evolved during the experiment may be brine, but the author prefers the use of a strong solution of CaCl<sub>2</sub>, sp. gr. about 1.37, as recommended by Schulze (1905).

From the moment at which the dough is divided into two pieces of 400 grams each, to the moment at which the dough-pieces are placed in bottles *F*<sub>1</sub> and *F*<sub>2</sub>, about 4 minutes will elapse. The drops collected in

<sup>2</sup> Sometimes, when starvation of the yeast does not occur, *b* will be found to be some few cc. smaller than *a*. This is possibly due to some slightly adverse influence of the strong maltose-solution on the activity of the yeast.

the graduated cylinders  $C_1$  and  $C_2$  during the first minute are rejected. Thus 5 minutes elapse before measurements start. Consequently, if the prescribed proofing period is  $t$  minutes, the time of measurement is  $t$  minus 5 minutes in order that the measurement finishes at the same moment as the proofing period would have terminated, if the dough had been proofed in a proofing cabinet.

## PART 2

### APPLICATION OF THE CONTROL-METHOD DESCRIBED IN PART 1 TO DIFFERENT BAKING-TEST-METHODS DESCRIBED IN THE LITERATURE

#### Material Used and Baking-Test-Methods

Three different baking-test-methods were examined. In the following discussion these methods will be referred to as Methods I, II, and III, respectively.

Method I is identical with a method described by Schnelle (1929) and used in an interesting and comprehensive study of different varieties of wheat grown in Germany. Method II is the A.A.C.C. basic method, Blish (1929). Method III is one of the methods used by Humphries (1920) during the investigations he carried out for the English Home-Grown Wheat Committee, i.e., the method referred to as "B. T. 2."

In examining these three baking-test-methods, two different flours, both milled in Danish commercial mills, were used. One of these, flour *M*, was milled from a lot of No. 1 Manitoba Northern wheat, whereas the other, flour *R*, was milled from a lot of "Barletta Russo" (Argentine wheat). The composition of these two flours will be seen from Table I. From this table it appears that the two flours differ widely with regard to their diastatic activity. Flour *M* had the highest diastatic activity, whereas the diastatic activity of flour *R* was low; by no means extremely low, however. None of the flours was chemically treated.

TABLE I  
COMPOSITION OF THE TWO FLOURS USED IN THE EXPERIMENTS

Flour Designation	Moisture	Crude Protein	Ash	Acidity as	Diastatic Activity <sup>1</sup>
	P.ct.	P.ct.	P.ct.	pH	
M	13.4	11.97	0.45	5.80	167
R	15.3	12.03	0.47	5.90	83

<sup>1</sup> Milligrams of anhydrous maltose produced by digestion of 10 grams of flour at 27° C. for one hour.

Further, every test was performed with two different qualities of compressed yeast—one has been called *O*, the other *F*. Yeast *O* was of

quite ordinary strength, probably corresponding in strength to most of the yeast qualities used in Europe and the United States of America. Yeast *F* was a stronger, faster yeast belonging to a type which is often preferred in England and which to some extent is also used in Germany.

### Results of Tests Using the Control Method

All the doughs made by the different methods from the different flours and yeasts were examined, using the control method described in Part 1. The results, i.e., the different values for the quantities *a* and *b*, are recorded in Table II. In all instances, where *b* is greater than *a* (i. e., where starvation of the yeast was established), the values of *a* and *b* are printed in heavier type. The detailed discussion of the results by the three methods follows.

TABLE II

CC. OF CARBON DIOXIDE EVOLVED FROM 400 GRAMS OF DOUGH DURING THE PROOFING PERIOD, USING DIFFERENT BAKING METHODS

Baking Method Employed	Pre-scribed Proofing Period (Minutes)	Pre-scribed Proofing Temperature	Carbon Dioxide Evolved in cc.							
			Flour <i>M</i>				Flour <i>R</i>			
			Yeast <i>O</i>		Yeast <i>F</i>		Yeast <i>O</i>		Yeast <i>F</i>	
			<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>
Method I	45	35° C.	520	535	855	875	<b>585</b>	<b>655</b>	<b>370</b>	<b>935</b>
	75		1020	1030	<b>1300</b>	<b>1555</b>	<b>815</b>	<b>1230</b>	<b>500</b>	<b>1170</b>
Method II	55	30° C.	520	530	<b>370</b>	<b>760</b>	<b>470</b>	<b>585</b>	<b>200</b>	<b>670</b>
Method III	25	85° F.	140	135	155	145	160	165	185	190

*a* is the amount of CO<sub>2</sub> evolved, maltose not being added.

*b* is the amount of CO<sub>2</sub> evolved, maltose being added to the dough.

### Results with Method I

The baking-test-method as used by Schnelle is as follows:<sup>3</sup> Dough composition: Yeast 4% (of the weight of the flour); salt 2%; water, according to absorption;<sup>4</sup> dough temperature 35° C.; punch after 30 minutes; mould dough after an additional 30 minutes. No fixed proofing-period is prescribed by Schnelle, but it is stated that the proofing-period may vary from 45 to 75 minutes. In the author's experiments, as will be seen from Table II, a 45 minutes proofing-period as well as a 75 minutes proofing-period was tried.

It appears from the data recorded in Table II, that starvation of the yeast occurred in all cases where flour *R* was used. The differences

<sup>3</sup> Here, and in the following discussion, only the essential features of the different processes can be rendered; for details the reader is referred to the original papers.

<sup>4</sup> In his experiments the author gave 58% water (of the weight of flour) to flour *M* as well as to flour *R*. Perhaps this amount of water was relatively a little too high for flour *R*.



between *b* and *a* vary from 655 cc. to 585 cc., or 70 cc., when yeast *O* was used; and from 1170 cc. to 500 cc., or 670 cc., when yeast *F* was used. When using the more diastatic flour *M*, yeast starvation occurred only at the longer proofing period of 75 minutes and where the stronger yeast *F* was used.

Undoubtedly the method used by Schnelle implies a large risk of starving the yeast, when the diastatic activity of the flour is relatively low. Consequently, the loaf-volume values obtained may, very often, be difficult to interpret, as the volumes of the loaves will be functions of strength as well as of the diastatic activity of the flour. The reason why yeast starvation will occur is not hard to find. As no fermentable sugar is added as a dough ingredient, the large amount of yeast used (4%) may soon exhaust the fermentable sugar available in the dough before the proofing-period is finished. Schnelle's method is an example of a baking method in which the dough-time and the dough-composition (especially the percentage of yeast) are not balanced against one another in a satisfactory way.

### Results with Method II

The A. A. C. C., Standard or Basic baking method, is as follows: Dough composition: Yeast 3% (of the weight of the flour); salt 1%; sugar 2.5%; distilled water 58%. All figures referring to flour are on 15% moisture basis. Dough-temperature is 30° C. First punch is made after 105 minutes, second punch 50 minutes after the first punch. Moulding takes place 25 minutes after the second punch. The proofing-period is 55 minutes. From Table II it will be seen that starvation of the yeast occurred in all cases where flour *R* was used. With flour *M* starvation occurred only when the yeast *F* was used. This means that the A. A. C. C. method may give unsatisfactory results as the volumes of the loaves will often be a function of the diastatic activity as well as of the strength of the flour.

As with Method I, it is apparent here also, that the amount of yeast and the dough-time are not properly balanced against one another. The results in Table II show that although 2.5% of sugar was added to the dough, a total dough-time of 235 minutes (180 + 55) is too long if 3% of yeast is used.

The practical importance of the phenomenon described, i.e., starvation of the yeast, is illustrated in Figures 2 and 3. These figures are photographs of loaves baked in absolutely identical manner according to the A. A. C. C. Basic Method. Exactly 600 grams of dough were scaled off for every loaf.

Figure 2 shows the results when yeast *O* was used. In the dough made from the flour *M* the yeast did not starve; in the dough made

from flour *R* the yeast starved slightly (see Table II). The loaf made from flour *R* although more voluminous than the loaf made from flour *M* would have been still larger if the yeast had not starved. Judging from Figure 2 one would conclude that *R* was the stronger flour.

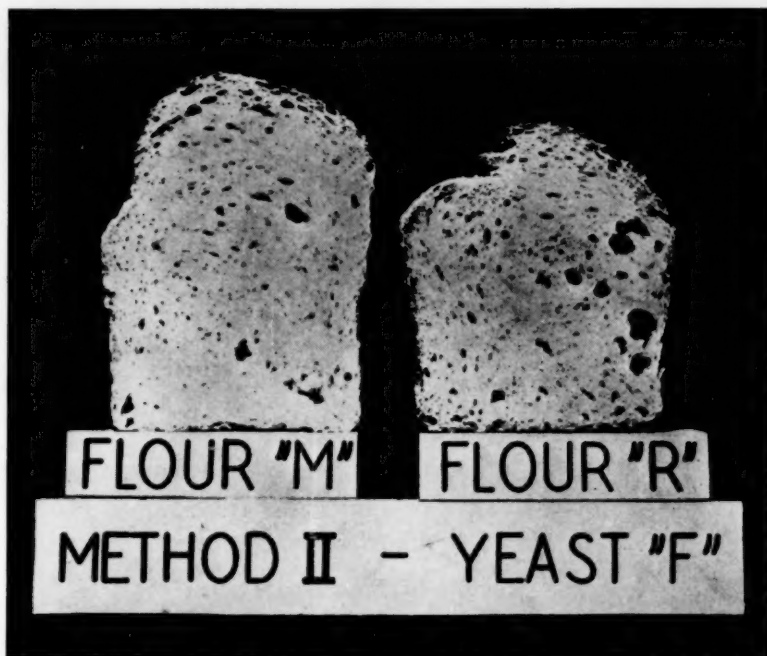


Fig. 3. Bread baked from flours *M* and *R* using Method II, and yeast *F*.

Figure 3 illustrates the results when yeast *F* was used. Here the yeast starved in the dough made from flour *M* (highest in diastatic activity) as well as in the dough made from flour *R* (see Table II). As will be seen from Table II, the starvation in the dough made from flour *R* was much more intensive than the dough made from flour *M*, as the amount of gas produced during the proofing-period was 1.85 ( $370 \div 200$ ) times the amount in the dough made from flour *M* as in the dough made from flour *R*. From Figure 3 it will be seen that flour *M* made the biggest loaf. Judging from Figure 3 one would conclude that *M* was the stronger flour, exactly the reverse conclusion one would derive from studying Figure 2.<sup>5</sup> Whenever "starvation" of the yeast occurs the volumes of the test-loaves become quite unreliable as indicators of the flour-strength.

<sup>5</sup> It may be of some interest to compare Figures 2 and 3 above, with Figures 1, 2, and 3 in Werner and Siedhoff's work (1929, p. 199 and 200). In the tests illustrated in Figures 2 and 3 the kind of flour as well as the kind of yeast were varied, whereas Werner and Siedhoff do not vary the kind of flour used but only varied the yeast.

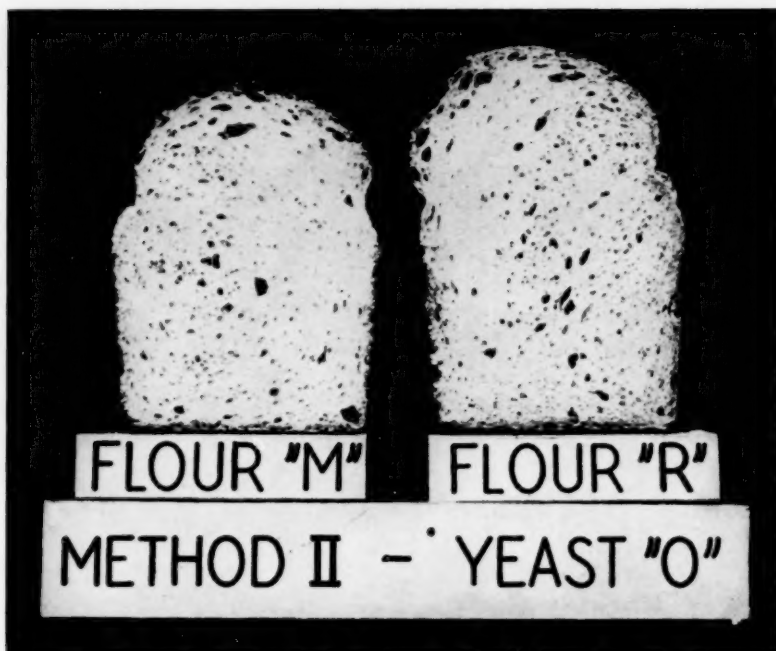


Fig. 3. Bread baked from flours *M* and *R* using Method II, and yeast *O*.

### Results by Method III

Humphries' "B. T. 2 method" is as follows: Dough composition: Yeast 0.75% (of the weight of the flour); salt 1½%; malt extract ("highly diastatic") 0.15%; water according to absorption;<sup>6</sup> dough-temperature, 85° F.; dough-time, 4 hours including the proofing period, which is prescribed to be "about 20 minutes." As will be seen from Table II, the author has used a 25-minute proofing period. The malt extract used in the author's experiments was a Danish commercial malt-extract.

It will be seen from the results recorded in Table II that Method III in all cases kept strength separated from diastatic activity; starvation of the yeast did not occur in any case. As practically the same total dough-time was used according to Method III as was used in the A.A. C.C. method (Method II), 240 minutes as against 235 minutes, it is evident, that it is the relatively small amount of yeast used (in connection, partly, with the addition of malt-extract) which accounts for the satisfactory results obtained by Method III. Method III thus offers an example of a method in which the dough composition and the dough time are well balanced against each other.

<sup>6</sup> In his experiments the author gave 53½% water (of the weight of flour) to flour *M* as well as to flour *R*. This amount of water was perhaps relatively a little too high for flour *R*.

### Discussion

It is the author's opinion, that the confusion undoubtedly existing concerning the question: "How to perform reliable baking tests?" might to some extent be avoided if care was always taken, when comparing the strengths of different flours, to use a baking method in which the possibility of yeast starvation (i.e., shortage on fermentable sugar) is precluded. In this connection it may be mentioned how this problem has been attacked in the investigations carried out, since 1929, by the author, for the Danish "Hvedeudvalget" i.e., Danish Home-Grown Wheat Committee.

During the first year of operation of this Committee (Jørgensen, 1930), 116 different flours were examined. The loaves were baked, according to this fixed procedure: Yeast 0.9% (quality *O*); salt 1¼%; no sugar or malt-extract; water according to absorption; dough-time 150 minutes; proofing-time 40 minutes; dough-temperature 30° C. Before any conclusions as to the relative strength of the flours were drawn from the volumes of the test loaves, three of the flours, those having the lowest diastatic activity were submitted to the test described in Part 1. In no case *b* was found to be greater than *a*, and not until this had been ascertained were any conclusions drawn as to the relative strengths of the flours.

As shown in Part 2, the A.A.C.C. method does not guarantee that a shortage on fermentable sugar will not occur. On the contrary, it was shown that such a shortage of fermentable sugar may occur when the diastatic power of the flour is relatively low, even if the yeast is of quite ordinary strength. When stronger yeasts are used the shortage of fermentable sugars will very frequently occur.

In the author's opinion the A.A.C.C. method would be a much more valuable one if the amount of yeast was reduced for instance from 3% to 1%, the method being otherwise unchanged.

The great influence of the quality of the yeast on the baking results as noted for instance in Figures 2 and 3 makes clear how important are the endeavours of the A.A.C.C. for creating a standard yeast testing procedure. Clark (1929), Werner and Siedhoff (1929), and Cook and Mallock (1930), have given important contributions to this question but the last word may not yet have been said about this problem.

Subjects related to that discussed by the author in this paper have often been treated in the literature and the author would, finally, like to comment on two papers recently published, that by Elion (1928), and that by Moen (1930).

Elion shows in a very clear manner, that under certain circumstances more carbon dioxide is developed from dough to which sugar

has been added than from corresponding doughs to which no sugar was added. However, from the work of Elion one cannot, but get the impression that Elion finds that the addition of sugar to the A.A.C.C. dough is somewhat unsafe as important flour properties such as its diastatic power might be obscured by the addition of sugar.

In direct contrast to Elion the author is of the opinion that it is a good feature of an experimental baking-method that the diastatic activity is obscured. In fact, following Humphries' principle, the diastatic activity must not influence the baking results at all. The author's objection to the A.A.C.C. Basic Method is quite opposite to Elion's. The author criticizes the A.A.C.C. Basic Method because *in spite of the addition of sugar* this method does not guarantee that the diastatic activity will not influence the baking result.

Moen (1930), expresses some doubt as to the utility of the A.A.C. C. method. To quote from his paper: "... it appears that the A. A. C. C. basic procedure has too severely taxed the diastatic capacity of Nos. 2, 3, and 5 . . ." and later on: "It is possible that some good qualities which these flours may possess have been obscured by a too strenuous fermentation process." The author agrees with Moen in this respect; the A.A.C.C. method will inevitably obscure good qualities which low-diastatic flours may possess. But in his summary in the same paper Moen nevertheless concludes: "In order to differentiate between flours varying in strength, a baking formula must have time and temperature so adjusted that diastatic capacity is severely taxed." Here the author does not agree with Moen; in the author's opinion *the diastatic activity should not at all influence the results of the baking test*. For determining the diastatic activity we dispose of excellent chemical methods (all the methods based upon Rumsey's fundamental principle) and we do not need expensive baking tests for being informed about the diastatic activity of a flour. On the contrary, the purpose of making a baking test should be to procure information about all those more or less well-defined physico-chemical flour-properties (stability, gas-retaining-power, etc.) which together constitute the "strength" in its proper sense. As is well-known, these last-mentioned properties have been extremely difficult to determine by analytical-chemical methods, and the use of expensive baking tests for determining these properties is well justified.

### Summary

1. It is emphasized, that in working out formulae for making baking tests care must be taken to make these formulae in such a manner, that the volume of the finished test-loaf will be a function of the "strength" of the flour only and *not* a function of the "strength" as well as of the



"gassing-power" (diastatic activity). The author thus agrees with Humphries' statement that "strength" and "gassing-power" are independent of each other and should be examined separately.

2. It is explained, that undesirable confusion of "strength" and "gassing-power" will occur if the yeast "starves" (i.e., suffers on account of shortage of fermentable sugar).

3. A technical control method is indicated, which permits of a decision as to whether in following a given baking-test-method one actually keeps "gassing-power" separated from "strength."

4. The control method mentioned in item 3 was applied to three baking-test-methods described in the literature. It was found that only one of the three methods was quite safe with respect to separating "strength" and "gassing-power."—The A.A.C.C. Basic Method was open to criticism in this respect. The author has emphasized that the amount of yeast used in the A.A.C.C. method is so high, that a separation of "gassing-power" from "strength" very often will not be obtained.

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### RELATION OF QUALITY IN DRY SKIMMILK TO BAKING STRENGTH<sup>1</sup>

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(Read at the Convention, May, 1931)

The amount of dry skim milk used by bakers is so steadily increasing year by year that the question of the baking quality of this ingredient should be of growing importance to Cereal Chemists. The enhanced popularity of milk solids as a constituent of bakers' products is largely due to an awakened public realization of the paramount importance of nutritive values in the selection of food. But increased nutritive value alone has not effected this higher rate of consumption of milk as an ingredient of the nation's bread. Important contributions toward this end have been made by the producers of dry milk. The baker who wished the advantage of milk solids in his product has been impressed by the convenience, economy and uniformity of supply that was made possible by dry milk. These advantages have been further augmented by the research and experimentation of dry milk producers which have resulted in a product that will appreciably improve bread quality when added as a dough ingredient.

Milk may be altered, both physically and chemically, by being subjected to heating. Since the production of powder from the fluid milk may involve the use of elevated temperatures for pasteurization and condensation, the industry has devoted considerable energy in investigating the effects of heat upon its product. These investigations have included studies made by various physical and chemical methods and will not be detailed here.

Our prime interest in methods for the production of dry milk lies in their effect upon the baking quality of the product. Scientists in the

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Bureau of Dairy Industry have been leaders in investigating the effects of heat upon milk as measured by its baking quality. In 1927, Greenbank, Steinbarger, Deysher and Holm demonstrated that if fluid milk was heated previous to being dried the product was improved from a baking standpoint and the viscosity of a water suspension of this milk powder was increased. This work was continued by Grewe and Holm and in 1928 they reported an improved product in respect to baking quality due to forewarming the fluid milk. The viscosity of the dry milk suspensions were higher as a result of such heat treatments but the viscosity and baking quality increases were not comparable in every case throughout the range of forewarming temperatures which were used in their work.

Further investigation of such preheating effects seemed desirable and one phase of the work involved the preparation of a series of five dry skim milks in a manner quite similar to that employed by Grewe and Holm (1928). These lots of powder were prepared from the same milk supply, in the same plant using a uniform processing procedure except for variations in heating treatment to which the fluid milks were subjected before being condensed and dried by a spray process. In preparation No. 1 the fluid milk was preheated as little as possible and temperatures did not exceed 52° C., and for only a few minutes. Preparations No. 2, 3, 4 and 5 were preheated for 30 minutes and at temperatures of 63°, 77°, 88° and 96° C., respectively.

Baking tests involving the use of 6% of each of these milks were made using a procedure essentially like supplement B of the A. A. C. C. baking test. Absorptions were varied and fermentation periods of 100, 140 and 180 minutes were allowed each of the three aliquots obtained after mixing each dough, using 300 grams of flour. A fairly strong, hard wheat, standard patent flour of medium baking quality was used in these comparisons. Each baking test was replicated six times.

Optimum baking results were obtained by the use of 100 and 140 minute fermentation periods with the milk-free doughs as well as from the doughs containing each of the dry milks under comparison. Differences in fermentation rate were not significant. In comparing baking qualities, a single figure baking strength score was used and was computed from the data for the 12 loaves containing each milk sample which had been fermented for 100 or 140 minutes.

The single figure score used was partly based upon the method of Larmour (1929) although only loaf volumes, grain and texture scores were included as factors, since these were considered to be the primary measures of baking strength. Loaf volumes in cubic centimeters, as obtained, were reduced by 300 and multiplied by 0.2, texture and grain

scores were multiplied by 1.0 and 2.0 respectively and the sum of these three results was taken as the index of baking strength.

The dough mixer used in these baking tests was equipped with a watt-hour meter attachment to accurately measure dough plasticities as described by Bailey (1930). In this manner it was possible to determine the amount of water required by each dough and to measure absorption effects of the various milks with considerable accuracy.

Before presenting the results of the baking tests, mention will be made of the methods used for determining the milk viscosities in order that the two sets of data may be compared. Viscosity methods were studied in attempting to establish such a procedure as a means of measuring baking quality in dry milk. Straight dry milk-water suspensions were selected as the most reliable for such comparisons. Torsion and flow type viscometers have both been used with little advantage for either except in the matter of ease of preparation of the suspension which favors the latter, since lower concentrations may be used with suitable viscometers of the Ostwald type than with an ordinary torsion instrument. In comparing viscosities of a series of dry milks, the flow type frequently has the added advantage that it may be used with any number of milks prepared in the same concentration while a torsion instrument is limited in range and sometimes cannot be successfully used in the same way on milk powders with wide viscosity differences.

The agreement between values obtained when measuring viscosities of the five dry milks in this series with Ostwald and MacMichael instruments was very good. Therefore only the Ostwald values are given. These results were obtained on suspensions of 20% dry skim-milk in water, mixed and held at 25° C. for two hours and determinations made using an instrument with a water equivalent of 140 sec./5.

Baking and viscosity results are given in Table I and are in part pictured in Figure 1.

TABLE I  
BAKING AND VISCOSITY RESULTS WITH DRY SKIMMILKS

Sample	Preheating treatment	Baking strength score	Increase in score over milk-free	Absorption (Increase over milk-free) %	Viscosity of dry skimmilk (Ostwald) sec./5
Milk-free		47.6			
Preparation 1	very little	53.7	6.1	4	395
Preparation 2	63° C., 30 min.	56.1	8.5	4	359
Preparation 3	77° C., 30 min.	62.2	14.6	4	401
Preparation 4	88° C., 30 min.	62.6	15.0	6	529
Preparation 5	96° C., 30 min.	62.2	14.6	6	504

It will be observed that preheating to 77° C. gave practically the maximum improvement as measured by baking strength scores. However, higher temperatures were needed to effect increased absorption and viscosity. These results (as well as others obtained in a study of heating fluid milks) indicate that lower preheating tempera-

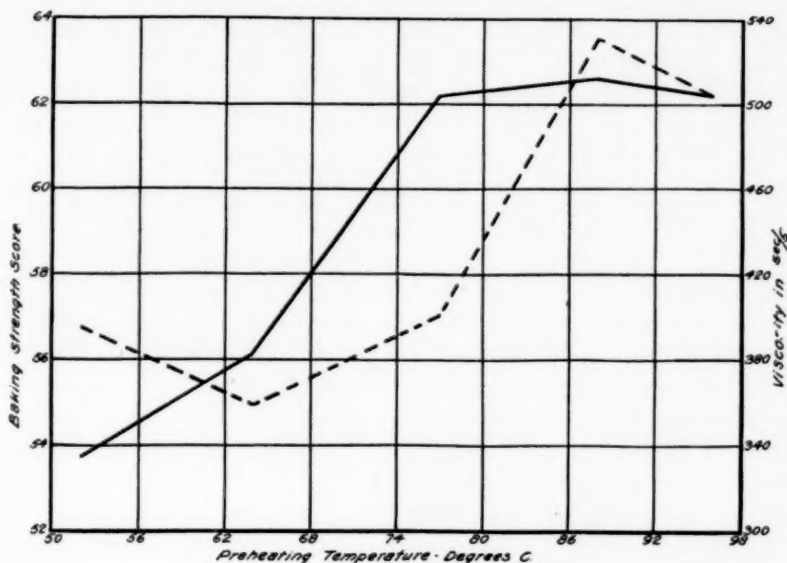


Fig. 1. Comparison of dry skim milk viscosities and baking strength scores.  
Solid line—Baking strength score.  
Broken line—Viscosity.

tures may be effective in increasing baking quality than are prerequisite to increased viscosity and higher absorption. Experience has shown that viscosity is often an indication of baking quality in dry skim milks. It appears that many of the exceptions which have been noted to the above generalization may be caused by the behavior of those milk powders which have been preheated to such intermediate temperatures that baking quality has been more decidedly improved than is indicated by the viscosity of a suspension of the dry milk.

Effect of time of preheating was compared by means of two samples of dry skim milk which had been preheated at 71° C. for 1 1/2 and 14 hours respectively; practically no difference in baking quality was found in these milks. A small viscosity and absorption increase was effected by the longer preheating. Time may not be as important as temperature in their relative effects upon fluid milk providing the other variable is kept at a low level. Certain results obtained with fluid milks appear to bear out this contention. Though no such generalization is proven by any single comparison, it appears probable that much



of the baking quality improvement had been effected by the 90 minutes preheating at 71° C. in this case and that subsequent heating at this temperature was chiefly reflected in the increased viscosity of the product.

It is apparent that the increase in viscosity does not necessarily parallel the improvement in baking quality which is effected by preheating. The colloidal changes, as determined by viscosity methods, are not a direct measure of the increases effected in the baking quality of milk by heat treatment. Other physical and chemical measurements have been made upon the dry milks of this series in attempting to throw some light upon the nature of the alterations or rearrangements which are effected by heat and which result in such decided improvements in quality from a baking standpoint.

Hydrogen ion determinations of these milks in water and flour-water suspensions revealed insignificant differences as a result of heat treatments except in the case of the sample which had received the 14 hour preheating. All of the other milks when added in 6% concentration (as compared to flour weight) reduced the hydrogen ion concentration of the flour-water suspension by 0.43-0.49 pH units as shown in Table II. The effects of the milks which had been preheated for 30 minutes at various temperatures were thus nearly the same and hydrogen ion changes cannot be considered as a cause of observed baking quality differences. The dry milk which had been preheated for 14 hours was 0.45 pH units more acid when in flour-water suspension than the corresponding milk with the 1 1/2 hour treatment but in this case changes in baking quality were negligible so that there appeared to be no relation between these characteristics.

TABLE II  
EFFECT OF HEAT TREATMENT UPON THE PH OF DRY SKIMMILK IN FLOUR-WATER SUSPENSION

Sample	Milk-free	D.S.M. <sup>1</sup> 1	D.S.M. 2	D.S.M. 3	D.S.M. 4	D.S.M. 5	D.S.M. preheated 1½ hrs. at 71° C.	D.S.M. preheated 14 hrs. 71° C.
ph	6.13	6.62	6.62	6.60	6.56	6.56	6.56	6.11

<sup>1</sup> Dry skim milk.

If changes in concentration of soluble minerals were a result of different heat treatments such effects should be measurable by electrical conductivity methods. Such measurements were made upon suspensions of low and high preheat milks of this series and also upon fluid milks which had been subjected to various degrees of heat treat-



ment. Heating fluid milks for 1 to 22 hours at 71° C. and for 30 minutes at temperatures varying from 63° to 96° C. effected only minor changes in their electrical conductivity. A 10% suspension of dry skim milk from preparation 1 gave an average specific conductivity value of  $6.31 \times 10^{-5}$  whereas a similar suspension from preparation 4 gave a reading of  $6.22 \times 10^{-5}$ . Thus the dry milk which had received the higher preheating was slightly lower in electrical conductivity than the other but the difference appears too small to be significant.

Saccharogenic activity of flour-water suspensions containing dry milks have also been studied in attempting to discover the specific cause of observed baking quality differences. Extensive observations have indicated that dry skim milk lowers the activity of diastatic enzymes when added to a flour-water suspension. This may be entirely due to decreases in the acidity of the suspension which would tend to inhibit this enzyme activity. With certain high diastatic flours this retarding effect of dry skim milk upon saccharogenic activity may be advantageous in that it limits the amount of sugar formed and makes possible the addition of more milk without excessive caramelization in the bread crust.

The effects upon saccharogenic activity of low and high preheated dry milks were compared allowing periods of digestion of from 1 to 3 hours before inhibiting enzyme activity. The differences which have appeared to date in such comparisons hardly seem large enough to justify the assumption that the observed baking quality differences of dry skim milks are due to effects upon the diastatic enzymes of flour.

The flour used in the baking comparisons of these milks was somewhat low in saccharogenic activity. In spite of this fact, increased loaf volumes were obtained through the use of each of these dry skim milks. Since milk reduces saccharogenic activity, which might be considered as detrimental with such a flour, it appears that this must be more than counter-balanced by other improving effects upon dough quality.

The nature of such effects is still unknown. Likewise it may be said that the nature of the changes which are effected by preheating dry skim milk as reflected in baking quality, are still undetermined. The facts of such improvement appear to be well established and some further light has been shed on the degree of heat treatment required to produce these changes.

### Summary

1. Heating skimmilks for 30 minutes at 77°, 88° or 96° C. before drying greatly improved the baking quality of the powder and to about the same degree at each of these temperatures.

2. The water absorption of the dough to which the dry milk was added and the viscosity of the milk powder in water suspension were not increased by preheating to 77° C. for 30 minutes. Marked increases were effected in these particulars by using the higher temperatures.

3. The use of viscosity methods in determining the baking quality of a dry milk will not give reliable results when dealing with products which have received intermediate preheating treatments.

4. Hydrogen ion concentrations of dry milks in flour-water suspension were not effected by these preheating treatments.

5. Electrical conductivity was only slightly different in water suspensions of dry milks having received a light and a severe preheating treatment.

6. Dry milks depress the saccharogenic activity of flour-water suspensions but this enzyme effect has not shown any significant agreement with temperature of preheat treatment. In spite of the inhibiting effect of dry skimmilks upon this enzyme activity, the use of milk improved baking quality in each case.

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## EVALUATING NEW WHEAT VARIETIES BY THE USE OF THE BAKING TEST

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### Introduction

The Agricultural Experiment Stations located in wheat producing areas, are continually developing new varieties. In order to replace older varieties, these new types of wheat should be better yielders, more resistant to plant diseases, and at least the equivalent in technical value of the wheats which they are to replace. In dealing with bread wheats baking quality shows greater variability and is generally deemed of greater importance than milling characteristics.

In the early stages of the development of new varieties only a small quantity of wheat is usually available for testing. One must, therefore, attempt to predict from laboratory tests, the future actual performance of the flour milled from the new wheat. Several years will usually elapse between the time when the wheat is selected for quality on the basis of laboratory tests, and the time when production assumes commercial importance. The prediction of quality from laboratory tests in this case may, therefore, be aptly termed a "long-distance forecast." The commercial mill on the other hand is able to almost immediately check laboratory findings against actual performances of their flour in the bake shop. This long distance forecasting, necessary in the case of new varieties, greatly increases the complexity of the problem and it is very essential that one observes and takes into consideration relatively small differences in baking characteristics.

It is now recognized that the results of the ordinary baking test as usually interpreted have distinct limitations as a means of evaluating quality. Present opinions regarding the value and use of the baking test reflect the results of comparatively recent investigations.

Weaver and Goldtrap (1922) published results of studies on baking tests, which seriously questioned the value of existing methods of judging flour strength. Weaver and Goldtrap used a series of flours which showed considerable variation in size of loaf when the same procedure was used for each flour; but when optimum conditions as to absorption, fermentation time and pH were provided for each flour, the loaf volume was practically the same. Harrel (1926, 1929) called attention to a number of variables in bread production, which will affect quality. Harrel called attention, in particular to the im-

portance of the time of fermentation. Clark (1929) presented data showing the reaction of different flours to prolonged fermentation.

The work of Blish (1927-28), Harrel (1929b), et. al. in developing the present standard A. A. C. C. baking tests, has further called attention to the importance of different variables, and has given a new conception of the value and use of the baking test. The A. A. C. C. standard test, using only 100 grams of flour, has a distinct advantage in testing new varieties. The amount of wheat available for test is limited and the use of 100 grams of flour as compared to 340 grams of flour as formerly used permits a larger number of baking tests from the same amount of material. Blish (1928b, 1929) also has given us definite standards for type of loaf and grain of crumb.

The A. A. C. C. standard procedure, further, rigidly fixes the conditions under which the baking test is made, and in this regard differs significantly from some other test baking procedures which it has replaced. This difference should be given consideration when interpreting results of baking tests. It is reasonable to presume, therefore, that results of baking tests by the A. A. C. C. procedure may require a somewhat different interpretation than has been previously employed.

In evaluating new wheat varieties, it is now customary to use a standard commercial variety, such as Marquis, for comparison or reference. At the North Dakota Experiment Station, it is the policy to select those new types, which most closely approach Marquis in baking characteristics. This practice is admittedly open to criticism, since such a method may eliminate types which might ultimately prove to be definitely superior to present accepted commercial varieties. On the other hand, such a method of selection, would serve to guarantee to the grower and to the miller, that the new wheat will be equivalent in quality to the type which it replaces. One should not overlook, however, the possibility and probability of producing wheats which will be distinctly superior to existing types in baking strength.

This paper will consider the criteria usually used in interpreting results of baking tests and in addition will also show results of classifying varieties according to type of loaf produced and fermentation tolerance.

### **Significance of Standards Now Used in Interpreting the Baking Test**

#### **CRUMB COLOR**

When considering color, we must distinguish between variations due to varietal characteristics and variations due to other factors. Damaged kernels, milling procedure, and the degree of extraction, will affect color, but these factors bear little relation to varietal character-

istics. The amount of yellow color present, however, is a distinct varietal characteristic.

Considering present market demands for bread flours, an excessive yellow color is objectionable. In our experience, however, the degree of pigmentation can be more satisfactorily determined by such tests as "gasoline color value," than by scoring the loaf. Objectionable types, carrying excessive yellow color, may, therefore, be eliminated through chemical tests, and the "color score" becomes of minor importance in evaluating varieties.

#### GRAIN OR POROSITY OF CUT LOAF

The use of the photographic standards proposed by Blish (1928) in scoring loaves is to be recommended. The following remarks are based on data secured during the past two years by the use of these standards. The grain or porosity of the cut loaf is no doubt important, but in evaluating varieties, the data would indicate that differences in grain serve only to distinguish wide differences in quality. The porosity of types in the questionable class does not always show significant variation from standard types, when the appreciable variation in grain of loaves from the standard varieties is considered.

A significant seasonal variation has also been noted. In the 1929 crop there was an appreciable spread in grain types. A number of questionable types in this case appeared to be below standard. In 1929, Garnet, Kota, Hope, Marquillo, Progress, Quality, Whiteman, and Marvel appeared to have a decided tendency toward a coarse open grain. In 1930, however, the only types showing this tendency were Progress and Marvel.

When a long fermentation period is used the better varieties show less deterioration in grain than others, but here, also, there is no sharp distinction. The loaf marks or "scores" cannot be used to differentiate between types showing relatively small differences in baking characteristics.

#### LOAF TYPE OR SHAPE

Blish (1928b) has suggested six types or shapes of loaves, designated as F, G, H, I, J, K. While the type of loaf is affected by the molding and panning, the same operator will tend to obtain the same types of loaves from a given flour. At the North Dakota Experiment Station, the loaves are molded comparatively tight. Flour from Marquis or standard spring patents under these conditions will have a strong tendency to give J or smooth type loaves.

Figure 1 shows the per cent of J type loaves obtained from a number of varieties over a period of two years, when baked by the standard

A. A. C. C. procedure. With the exception of Ruby, Preston, and Progress, the data is based on 24 bakings.

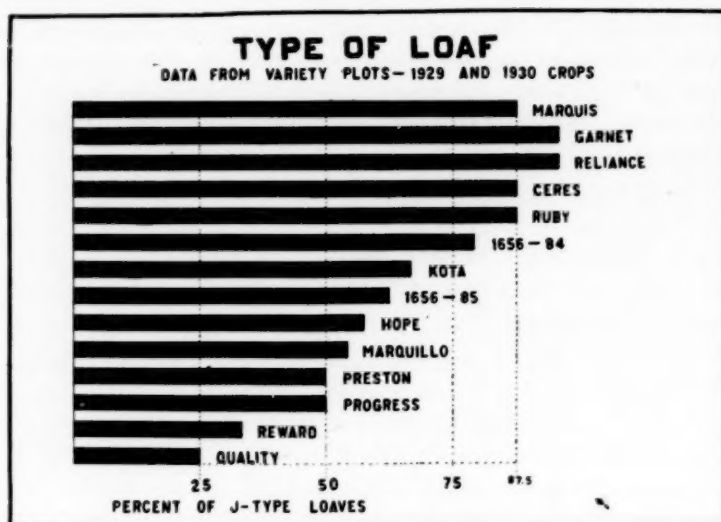


Fig. 1. Type of loaf.

Marquis and certain other varieties it will be noted show a decided tendency to produce the smooth or J type loaf. It will be noted that Garnet, Reliance, Ceres, and Ruby have as great or greater tendency to produce as smooth loaves as Marquis. Preston is generally considered to be of poor quality, and varieties showing similar classification as regards type of loaf are Quality, Reward, Progress, Marquillo, and Hope. The Quality, Reward, Preston, and Progress varieties produce the smallest number of smooth loaves. These four varieties, under the molding practice in this laboratory, usually produce the F type loaf. Kota, and the two North Dakota selections (1656-84 and 1656-85) are intermediate.

The type or shape of loaf is affected by the molding and panning procedure. In order to eliminate the personal equation, a number of observations are necessary in order that one may obtain reliable data. This method of classifying varieties would appear to have real value and deserves further study.

Why should the type of loaf be an important consideration in judging quality? When molded in the same manner, it appears reasonable to suppose that a strong tough gluten would be more likely to give a J type loaf than the F, H, or G types. In case of the smooth or J type loaf, the gluten has had sufficient strength to resist the gas pressure, which causes the break and shred in the other types. The type of



loaf, therefore, probably is a very important criterion of quality, and one is inclined to regard with suspicion those varieties which do not tend to produce a large percentage of smooth or J type loaves.

#### LOAF VOLUME

What is the relation between loaf volume and baking strength? Large loaf volume and high baking strength were, a few years ago, considered synonymous terms. Recent investigations, however, have to a considerable degree shaken our confidence in loaf volume as a final index of baking quality. If one were to rank new wheat varieties as to baking strength solely on basis of the loaf volume obtained by the ordinary baking test the conclusions regarding their baking quality would in many cases be incorrect. Recent observations in this laboratory tend to indicate that a large loaf volume may not necessarily indicate high baking strength. Size of loaf is the result of the effect of several factors, not all of which have the same relation to baking strength.

*Loaf Volume and the Type of Loaf.*—A serious error in using the size of loaf as an index of baking quality is the fact that we are often comparing the volume of loaves of quite different type or shape. Varieties which tend to give F or H type loaves, usually average higher in volume than varieties which give the smooth or J type loaf.

In making duplicate baking tests on samples, the two duplicates usually are the same type of loaf. Occasionally, however, two different type loaves will be produced on duplicate bakes. The most frequent variation in our laboratory is between the J and F type. In going over baking data, 35 instances were found in which these different type loaves were recorded for duplicate bakes. The F type loaves averaged 28 cc. higher in volume, and in every instance except one the F type exceeded the J type in loaf volume. The maximum variation in this series was 62 cc. This difference in loaf type and the resulting differences in loaf volume are due no doubt to differences in molding and panning.

Loaf volume, therefore, might be a more reliable index of baking strength if it were always possible to compare the size of loaves of the same general type. When the type of loaf varies, however, a larger volume may not be indicative of superior baking strength, and may actually indicate a weakness in the gluten.

*Loaf Volume and the Baking Procedure.*—The volume of loaf is comparatively sensitive to any change in the test baking procedure. It is recognized that the direction and magnitude of such changes vary with different flours. The variation of loaf volume with variation of baking procedure, in our judgment may be very useful in classifying and evaluating new wheat varieties.

### Experimental

During the past two years a series of variety samples have been baked by four different procedures as follows: (1), the standard A. A. C. C. procedure, (2), the standard procedure plus 1 milligram of potassium bromate, (3), fermentation time 1 hour shorter than standard procedure, (4), fermentation time 1 hour longer than the standard procedure. In 1929, samples from the variety plots at Fargo, Dickinson, Langdon, Williston, Edgely, and Hettinger were used for these studies. In 1930, samples from the Fargo, Langdon, and Williston plots, only, were used. These wheats averaged quite high in protein. The average of 116 samples from the 1929 crop was 15.2%, while the 61 samples from the 1930 crop averaged 14.9% protein. The 116 samples represented 33 varieties in 1929, while the 61 samples in 1930 represented 25 different varieties.

What is the relation between the results obtained by the standard baking procedure and the three supplementary tests?

The correlation between the loaf volumes obtained by the standard baking procedure and each of the three supplementary tests was determined. A statistical analysis of the data obtained will be found in Table I. The coefficients of correlation show considerable seasonal

TABLE I

STATISTICAL ANALYSIS OF LOAF VOLUMES OBTAINED BY DIFFERENT BAKING METHODS

Type of analysis	1929 <sup>1</sup> Crop	1930 <sup>1</sup> Crop
Number of samples . . . . .	116	61
Average loaf volume, standard procedure, cc. . . . .	452	438
Average loaf volume, bromate modification, cc. . . . .	447	442
Coefficient of correlation, standard vs. bromate . . . . .	+0.473	+0.873
Probable error of coefficient of correlation . . . . .	±0.048	±0.031
Average loaf volume, short fermentation method, cc. . . . .	460	478
Coefficient of correlation, standard vs. short method . . . . .	+0.536	+0.835
Probable error of coefficient of correlation . . . . .	±0.044	±0.039
Average loaf volume, long fermentation method, cc. . . . .	368	365
Coefficient of correlation, standard vs. long method . . . . .	+0.102	-0.103
Probable error of coefficient of correlation . . . . .	±0.062	±0.127

<sup>1</sup> 1929 samples from Fargo, Dickinson, Langdon, Williston, Edgely, and Hettinger plots. 1930 samples from Fargo, Langdon, and Williston plots.

variation, but they indicate a close relationship between the standard procedure results, and the results obtained by either the bromate or short modifications.

The loaf volumes obtained by the bromate modification averaged 5 cc. less than the standard in 1929, and 4 cc. greater in 1930. Considering the character of the crops, a small difference is to be expected.

In 1929, the coefficient of correlation between volumes obtained by the standard method and those obtained by the bromate modification was  $+0.473$ , while in 1930 the coefficient was  $+0.873$ .

The loaf volumes by the short modification averaged somewhat higher than those obtained by the standard procedure, but the coefficients of correlation are about the same order as those between the standard and the bromate modifications.

The long fermentation method as would be expected gives a much lower average loaf volume, and the coefficients of correlation are quite different. In 1929, the coefficient of correlation was  $+0.102$  while in 1930, the coefficient was  $-0.103$ .

These data would indicate that neither the bromate modification nor the short fermentation method can be expected to give a ranking of varieties which would differ materially from the ranking in loaf volume by the standard procedure.

In case of the long fermentation, however, there is apparently much less relation between the loaf volumes obtained by the two methods. In 1930, the correlation was negative. This indicates that the long fermentation procedure will give a quite different ranking of varieties, as regards loaf volume, than the standard procedure. The long fermentation modification, therefore, should be a more useful supplementary test than either the short fermentation method or the bromate modification.

#### Variation Due to Bromate Modification

Larmour and McLeod (1929) state "that the basic formula plus .001 per cent  $KBrO_3$  gives a more accurate estimate of the baking values of flours, since it more nearly approximates commercial practice." The bromate modification does, no doubt, give an indication of the tolerance of the flour to bleaching agents. As pointed out by Larmour and McLeod it may also indicate weakness due to damaged grain. The evaluating of varieties, is, however, a different matter.

The bromate modification does not appear to be of much service in evaluating hard spring wheat types. In Table II loaf volume measurements obtained by the standard procedure and by the bromate modification for a series of varieties grown on Fargo plots are compared. Apparently neither the total volume nor the differences in volume by the bromate modification, are reliable indices of variety quality.

If the varieties are ranked according to the volume obtained by the bromate modification, then Quality heads the list with Kota, Preston, 'Whiteman,' and Reward following in the order named. From other considerations the first two varieties would be classified as questionable, and Preston and 'Whiteman' as of poor quality.

TABLE II

COMPARISON OF LOAF VOLUMES BY STANDARD METHOD AND BROMATE MODIFICATION  
VARIETIES FROM FARGO PLOTS—AVERAGE FOR CROP YEAR 1929 AND 1930

Variety	A	B	Difference B-A
	Standard Method Average Volume	Bromate Modification Average Volume	
	cc.	cc.	cc.
Marquis	404	392	-12
Ceres	428	417	-11
1656-48	421	429	+ 8
1656-84	415	403	-12
1656-85	450	438	-12
Kota	505	529	+24
Garnet	426	420	- 6
Reliance	409	390	-19
Quality	486	538	+52
Marquillo	491	458	-33
Hope	452	404	-48
Reward	533	505	-28
Preston	476	515	+39
Progress	446	484	+38
Marvel	457	423	-34
Whiteman	497	509	+12

Quality, Preston, Progress, and Kota give a high positive reaction. Is this indicative of good baking quality? From other considerations we would rate these four varieties as either of questionable or poor baking quality. Hope, Marquillo, Reward, and Marvel show the largest negative reaction. Marquis, Ceres, and Garnet show a smaller negative reaction. Should one condemn varieties showing a negative reaction? The bromate supplementary test evidently does not help much in differentiating between good and poor varieties.

#### Volume Variation Due to Fermentation Period

As suggested previously, if we ranked varieties as to quality, on the basis of loaf volume secured by the ordinary baking test, our conclusions might be erroneous. If we take, for example, the series of varieties in Figure 2 we find that in this case, Marquis has the lowest volume by the standard A. A. C. C. baking test. Heading the list, we find Reward and Kota, and near the top we find such varieties as Marquillo, Quality, 'Whiteman,' and Preston. All of these varieties, however, give a relatively low percentage of J or smooth type loaves as compared to Marquis, and as previously noted, we are, therefore, comparing volumes of loaves which differ in type or shape.

When the fermentation period is varied, however, one gets a different picture. The "fermentation tolerance" or consistency of performance shows considerable variation. With a long fermentation period, some types will show a much greater decrease in volume than others.

This is shown in Figure 2. It is recognized that decrease of volume on long fermentation is due partly to lack of sugar, but this apparently is only one factor causing decrease in some varieties.

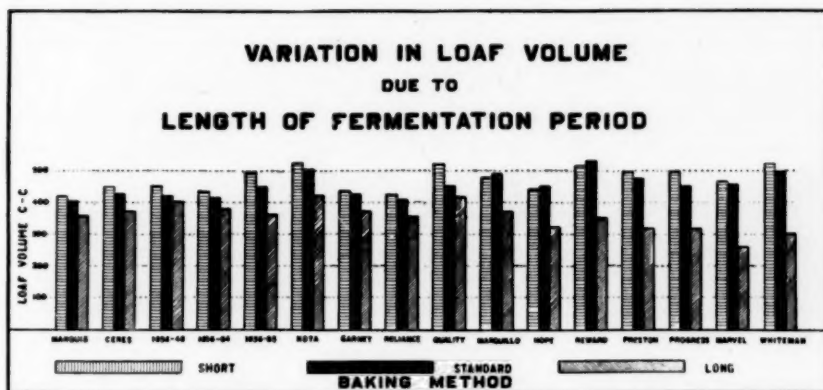


Fig. 2. Variation in loaf volume due to length of fermentation period.

Marquis shows a relatively small variation in loaf volume due to variation in fermentation time. In the same class with Marquis we find Ceres, 1656-48, 1656-84, Garnet, and Reliance. At the other extreme we find 'Whiteman,' Marvel, Progress, and Preston, and we were surprised to find Reward in the same class. Kota, 1656-85, Quality, Marquillo, and Hope may be classified as intermediate. Which is the most desirable to the baker, consistency or magnitude of loaf volume?

In making these differential tests we find considerable variation in different samples of the same variety or type, but such data tells us something that the ordinary baking test does not. Let us take for example the 'Whiteman' variety, which shows a variation of over 200 cc. in loaf volume due to fermentation time. Baked by the ordinary standard method this wheat almost invariably gives a good loaf of bread. The wheat is a semi-hard type, showing an excessively high protein content, and if loaf volume on the standard test were the sole criteria of quality it would be judged an excellent bread wheat. After noting the reaction of this flour to long fermentation, however, and other peculiarities, it would certainly not be advisable to recommend the extensive production of such wheat.

The variation of volume, therefore, with fermentation time, may be used as a measure of the fermentation tolerance of the flour. A satisfactory volume, with consistency of performance is presumed to be more satisfactory than a high volume obtained only under optimum conditions.



### Discussion

When evaluating wheat varieties, it should be recognized that a number of good points do not necessarily out-weigh a single important deficiency in quality. For example, a poor color or low absorption may definitely disqualify a variety which is entirely satisfactory in other respects. A good flour, further, must be capable of producing satisfactory bread under a wide range of bake shop practice. The type of loaf produced in test bakes, and the consistency of performance under varying fermentation time would appear to be important criteria of baking strength, when using the A. A. C. C. procedure and supplementary tests.

In using as a basis of judgment the external type of loaf and the variation of loaf volume with fermentation, we are able to classify varieties more satisfactorily. Accepting Marquis as a standard, we have a variety which has a strong tendency to give smooth or J type loaves, the color and grain are satisfactory, the loaf volume is not as large as in case of some other varieties, but the loaf volume is comparatively consistent and tends to show relatively small variation due to change of fermentation time. We then have varieties which are unquestionably poor varieties. Preston has never met with approval commercially, and Progress is a poor type. Preston, as compared to Marquis, has a definite tendency to give a low percentage of J type loaves, and the loaf volume shows considerable variation with fermentation. Progress shows a similar tendency.

Now we come to the largest and most difficult class—the questionable variety. Let us consider two varieties which have been placed on the market and have not met with entire approval—namely, Kota and Quality. Kota has a yellow color, but it also has in common with Quality two other characteristics, namely, the tendency to give less J type loaves and relatively less consistency in loaf volume as compared to Marquis. The variation in loaf volume is not as great, however, as in the case of Preston and Progress.

Other promising new varieties which this classification would place on the questionable list are Marquillo, Hope, and Reward. Marquillo has an objectionable amount of yellow color, but all three varieties show a relatively wide variation in loaf volume or in other words a limited fermentation tolerance. They also have a tendency to give a small percentage of J type loaves. We realize that in placing Reward in this class, our classification may be in disagreement with many laboratories. Reward has some other good points, but would come under this classification since it shows a lack of fermentation tolerance and tendency to produce less J type loaves.



The Ceres, Garnet, and Reliance types appear to be quite similar to Marquis in fermentation tolerance and type of loaf. Although Garnet is low in color this classification as to baking strength would appear to be in accord with the recent report of Newman (1930).

### Summary

1. The type of loaf and consistency of volume with varying fermentation time are suggested as methods of classifying hard spring wheat varieties as to quality.
2. Varieties have been classified according to tendency to produce J type loaves.
3. F type loaves will average higher in volume than the smooth or J type loaves.
4. The bromate modification of the baking test apparently does not have much value for classifying spring wheat varieties.
5. The variation of volume with fermentation time as a measure of fermentation tolerance gives a useful classification of varieties.

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**WHEAT AND FLOUR STUDIES XIX**  
**STUDIES OF THE EFFECT ON THEIR BREAD-MAKING**  
**PROPERTIES OF EXTRACTING FLOURS WITH ETHER,**  
**WITH SPECIAL REFERENCE TO THE GAS RETAINING**  
**POWERS OF DOUGHS PREPARED FROM ETHER-EX-**  
**TRACTED FLOURS<sup>1</sup>**

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(Received for publication May 21, 1931)

The improvement of the baking quality of flour by extraction with ether has recently received attention by several investigators. It was shown by Johnson (1928) that bread of better color, texture and volume was produced from ether-extracted flour than from the natural flour. This result was attributed to the removal of phosphatides, which substances, according to Working (1924), have deleterious effects on the bread making properties of flours. Recently Geddes (1930) has confirmed these results on the effects of ether-extraction and has found that the deleterious effects of certain germ constituents, presumably phosphatides, may be reduced, not only by partial removal from the flour by ether-extraction, but also by oxidation with chemicals or by heat. Working (1928a-b) also noted that phosphatides may be changed by oxidizing agents in such a way as to affect materially the baking properties of the flour and suggested that there may be an optimum phosphatide content, the presence of larger or smaller quantities of which will result in the production of poor bread.

Treloar, Sherwood, and Bailey (1931) found a significant positive correlation between crumb color and crumb texture of bread,

$$r = + 0.446 \pm 0.030,$$

from which it is evident that high color score is in general associated with high texture score. Since ether-extraction of flour results in improving the crumb color of the bread due to the removal of pigmented substances (which substances affect the color score, a factor associated with texture), the possibility exists, that the removal of anything which affects the color score, will also affect the texture score.

Mohs (1915, 1924) has studied the effects of ether extraction on the bread-making properties of flours and obtained greater volume and finer grained texture for the extracted flours. It appears, however, that he does not desire the fine grained texture and considered bread

<sup>1</sup> Contribution from the Chemistry Department, University of Montana, Agricultural Experiment Station, Bozeman. Paper No. 5, Journal Series.

from the extracted flour unsatisfactory on this account. The crumb of the bread prepared from the ether-extracted flour was less elastic than that of the bread prepared from natural flour. This property made the bread from the ether-extracted flour less desirable. In conclusion Mohs (1924) states that the fat contained in flour is necessary for the formation of the gluten, is adsorbed on the protein, and is not removed by washing with water, and also that the removal of fat from flour operates to prevent hydration. By fat, Mohs means the materials removed by extraction with ether.

It appears to us that Mohs' experimental results are in agreement with our own since finer grained texture and greater volume were obtained in both cases. There exist, however, differences in interpretation of the data which are due in part to differences in the types of bread desired in Germany and in the United States. In order to be able to understand more exactly the reasons why ether-extraction resulted in producing flours capable of yielding bread of better texture and volume the work reported in this paper was conducted.

### Experimental

#### BAKING CHARACTERISTICS OF NATURAL AND ETHER-EXTRACTED FLOURS

A series of freshly milled flours consisting of a 3d middlings, a straight, a 1st clear and a 2d clear flour were extracted for 72 hours using a Schmidt extractor (1916). After extraction the ether was allowed to drain off and the flour spread out in a thin layer, the ether being allowed to evaporate at room temperature. When free from ether the flours were stored in glass jars until required for use. The pH, ash, protein, diastatic activity and baking data on these flours are given in Table I. The baking test used for this work was that described by Sharp and Whitcomb (1926), equal quantities of flour, on the 13.5 per cent moisture basis, being used. The results obtained for the baking test are in good agreement with those of Johnson (1928), the breads prepared from the extracted flours being higher in their volume, color and texture scores than those prepared from the natural flours. Thus the volume, the color and the texture scores for the extracted 3d middling flour were 2210 cc., 102 and 99; and for the natural flour, 2170 cc., 97 and 96 respectively.

Mohs (1924) stated that the ether extractable substances in flour were necessary for the formation of gluten. Our results do not confirm this statement. Satisfactory glutens were obtained from both natural and ether extracted flours and the quantity of gluten (wet or dry) was the same for the extracted as for the corresponding natural flour.

TABLE I  
CHEMICAL AND BAKING TESTS OF NATURAL FLOURS, ETHER-EXTRACTED FLOURS, AND THE SAME ETHER-EXTRACTED FLOURS CONTAINING ADDED FAT

No.	Description	pH	Ash dry basis (per cent)	Protein (N×5.7) dry basis (per cent)	Diastatic activity (Rumsay Units)	Loaf volume cc.	Color score	Texture	Absorp- tion
1	Natural 3d middlings flour	5.83	0.42	11.77	152.2	2170	97	96	68
1a	Ether-extracted 3d midds. flour	5.88	0.42	11.73	185.9	2210	102	99	70
1b	Ether-extracted 3d midds. flour, plus 3 per cent of lard					1750	100	98	70
2	Natural straight flour	6.09	0.61	12.60	141.7	2230	95	90	68
2a	Ether-extracted straight flour	6.24	0.61	12.59	225.4	2350	99	92	71
2b	Ether-extracted straight flour, plus 3 per cent of lard					2010	98	96	70
3	Natural 1st clear flour	6.22	0.90	14.08	140.5	1970	90	93	68
3a	Ether-extracted 1st clear flour	6.32	0.90	14.02	233.9	2040	96	93	69
3b	Ether-extracted 1st clear flour, plus 3 per cent of lard					1800	93	93	69
4	Natural 2d clear flour	6.26	1.58	13.53	211.8	1240	50	poor	64
4a	Ether-extracted 2d clear flour	6.44	1.60	13.95	354.5	1350	55	poor	66
4b	Ether-extracted 2d clear flour, plus 3 per cent of lard					1250	55	poor	72

Gluten puffs of about the same volume were obtained from natural and extracted 3d middlings and straight flours, but for the 1st clear and 2d clear flours gluten puffs from the extracted flours had considerably greater volumes than gluten puffs from the corresponding natural flours.

#### GAS RETAINING ABILITY OF DOUGHS FROM NATURAL AND ETHER-EXTRACTED FLOUR

In this work we were not so much interested in the baking data as in the mechanism which made it possible to produce bread of superior texture and volume from ether-extracted flours, an observation which has been confirmed by several investigators. It was thought that a study of the gas retention of the doughs would be of importance in this connection. The apparatus described by Bailey and Johnson (1924) was used for this purpose. By the use of this apparatus it was possible to measure the increase in volume of the dough, the increase in volume of the dough plus the  $\text{CO}_2$  lost from the dough and by difference to obtain the  $\text{CO}_2$  lost from the dough. Dough, equivalent to 50 grams of the flour, was placed in the apparatus and the progressive increase in volume noted at ten minute intervals. The data, which were obtained, are given in Tables II and III and the volumes occupied by the dough are shown graphically in Figure 1. A study of the data indicates that the extraction of the flour with ether had a marked effect on the gas retaining power of the dough prepared from it. Thus, after fermentation for four hours, the increases in volume of doughs prepared from 50 grams of natural and ether-extracted flours were 186 and 343 cc. for the 3d middlings flour, 135 and 250 cc. for the straight grade flour, 121 and 241 cc. for the 1st clear flour, and 86 and 133 cc. for the 2d clear flour respectively. Obviously the  $\text{CO}_2$  lost from the dough was considerably less for the doughs prepared from ether-extracted flours especially as in most cases the sum of increase in dough volume and expelled gas was lower for ether-extracted than for natural flour doughs. Thus ether-extracted and natural 3d middling flour doughs lost 84 and 237 cc. of  $\text{CO}_2$  respectively from total increases in volume which were practically the same.

The doughs which were placed in the apparatus were fresh and had not been subjected to previous fermentation. Since dough is allowed to rise several times (the gas being worked out at the end of each rise) before it is placed in the proofing cabinet and allowed to rise for the last time before baking, it was thought desirable to subject a dough to the regular bread-making procedure and then at the beginning of the proofing stage to place a portion of it in the Bailey-Johnson apparatus. Doughs made from natural and ether-extracted

TABLE II  
INCREASE IN VOLUME OF THE DOUGH AND INCREASE IN THE VOLUME OF THE DOUGH PLUS THE VOLUME OF CO<sub>2</sub> LOST FROM THE DOUGH FOR DOUGHS PREPARED FROM A 3RD MIDDINGS FLOUR. THE DATA ARE GIVEN FOR NATURAL AND ETHER-EXTRACTED DOUGHS, FOR SIMILAR DOUGHS PREVIOUSLY FERMENTED TO THE PROOFING STAGE AND FOR THE EXTRACTED FLOUR DOUGH CONTAINING 3 AND 5% OF ADDED LARD

Time (min.)	3d middlings natural		3d middlings ether-extracted		3d middlings fermented to proof stage		3d middlings ether-extracted proof stage		3d middlings ether-extracted, plus 3 per cent of lard		3d middlings ether-extracted, plus 5 per cent of lard	
	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.
10	0	0	0	0	0	0	0	0	0	0	0	0
20	11	10	16	9	7	8	7	11	10	8	8	8
30	21	23	33	20	18	18	15	27	23	25	21	21
40	45	41	48	32	27	28	24	43	41	40	37	37
50	63	58	60	44	37	37	33	57	50	53	48	48
60	80	73	76	54	46	46	41	73	65	70	63	63
70	103	93	102	65	55	57	50	97	86	95	88	88
80	125	115	122	73	62	65	57	115	105	115	105	105
90	145	128	143	85	72	74	65	133	118	132	122	122
100	170	137	167	94	77	82	72	155	133	153	132	132
110	190	137	186	103	85	90	80	175	142	175	133	133
120	208	143	205	112	91	98	86	195	152	195	135	135
130	230	148	227	122	98	106	93	217	155	218	132	132
140	250	158	245	130	102	113	100	233	158	235	135	135
150	273	168	271	140	109	121	107	253	165	255	137	137
160	286	166	285	150	113	130	114	271	170	271	137	137
170	305	170	303	158	117	137	119	290	163	290	138	138
180	325	173	323	167	121	144	126	308	173	307	140	140



190	341	178	345	296	174	121	151	130	327	176	323	140
200	356	178	358	310	182	123	157	135	345	172	341	142
210	373	176	375	320	189	126	163	140	360	178	358	142
220	390	185	393	327	199	125	171	145	378	176	375	143
230	406	185	410	338	202	128	178	149	395	180	392	145
240	423	186	427	343	207	129	182	149	412	180	407	142

3d middlings flours were therefore prepared and allowed to ferment. After the fourth rise, portions of the dough equivalent to 50 grams of the original flour were placed in the Bailey-Johnson apparatus and the test conducted in the usual way. The results given in Table II show that the dough prepared from ether-extracted flour again attained the greater volume but that the difference between the increase in volume for the two doughs was much less than when fresh doughs were used. Also the natural flour dough lost considerably more  $\text{CO}_2$  during the four hour fermentation than did the extracted flour dough. Thus the volumes attained by ether-extracted and natural flour doughs were 149 and 129 cc. and the volumes of  $\text{CO}_2$  lost from the doughs were 33 and 78 cc. respectively. It is therefore apparent that the extraction of flours with ether markedly increased the ability of doughs prepared from them to retain the  $\text{CO}_2$  produced during fermentation.

It is interesting to note that the quantity of  $\text{CO}_2$  produced by fermentation was much less when dough previously fermented to the proofing stage was used than when fresh dough was used. Thus for the 3d middlings natural flour the volume of dough plus volume of  $\text{CO}_2$  lost from the dough was 423 cc. for the fresh dough and 207 cc. for the dough taken at the proofing state. Similar results were obtained for the dough prepared from ether extracted flours.

Also the effect of allowing the dough to rise several times with "punching" at the proper time was to decrease the volume to which it finally rose during proofing. Thus in the case of natural 3d middlings flour the increase in volume was 186 cc. for the fresh dough and 129 for the same dough at the proofing period. In the case of the ether-extracted flour the increase in the volume was 343 cc. for the fresh dough and 149 cc. for the same dough at the proofing period. However, these larger increases in dough volume in the case of fresh doughs were obtained as a result of the production of considerably more carbon dioxide. At equal levels of increase in dough volume plus loss of  $\text{CO}_2$  from the dough the fresh and proof stage natural flour doughs attained volumes of 143 and 1

cc. respectively, while for the ether-extracted flour the volumes attained by fresh and proof stage doughs were 178 and 148 cc. respectively. Thus it is obvious that on this basis of comparison the pre-

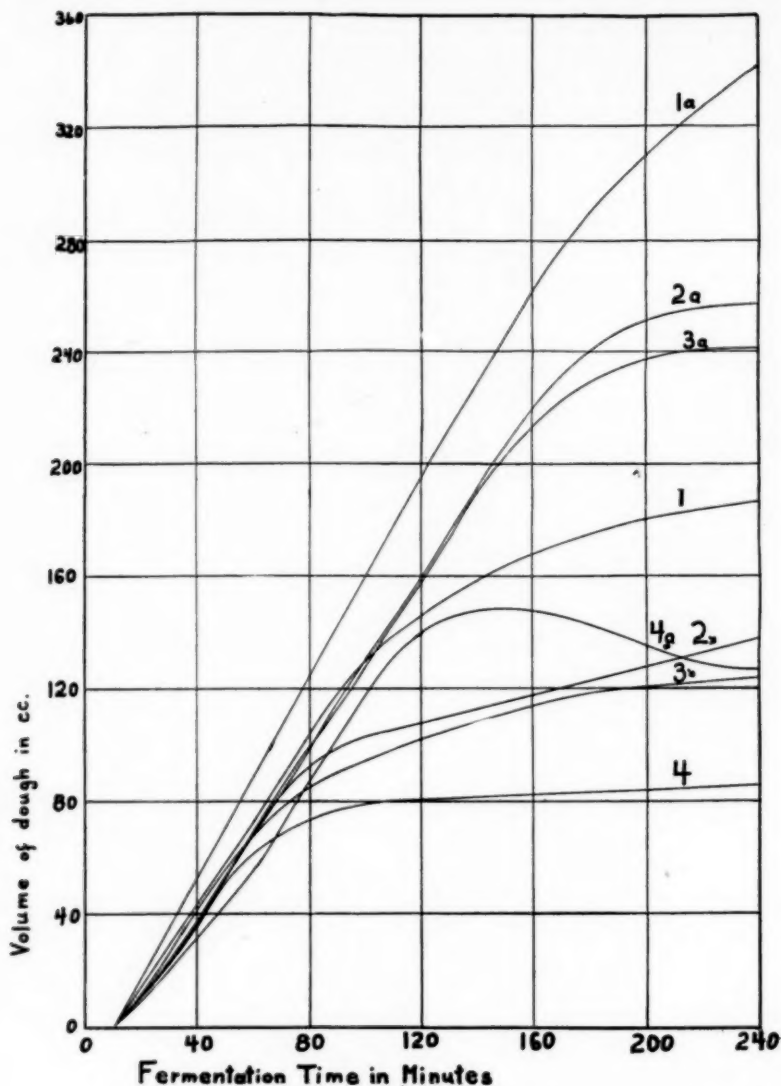


Fig. 1. Volumes attained by doughs prepared from natural and ether-extracted flours of several flour grades. Grade and chemical composition of the flours are given in Table I.

viously fermented doughs were also inferior to fresh doughs in their gas retaining powers.

### INFLUENCE OF FAT ON THE GAS-RETAINING POWER OF ETHER-EXTRACTED FLOUR

Since ether extraction of flours had such marked effects on their gas-retaining powers it was thought of interest to add a fat to the extracted flours and to note the effect of such addition on the gas-retaining power. Lard to the extent of 3% was therefore added to each of the extracted flours. In the case of the 3d middlings flour 5% of lard was also added. The results of the baking tests are given in Table I. The effects of the added lard on gas retention were determined only for the 3d middlings flour and the results are given in Table II.

The results of the baking tests as given in Table I show that the addition of lard to the extracted flours effected a decrease in the volume of the bread baked from these as compared with the volume of the bread baked from ether-extracted flour not containing the added fat. In the previous paper (1928) it had been found that the addition of 5% of lard to ether-extracted flour only slightly reduced the loaf volume. The results of the present work are therefore not in agreement with the earlier work as regards the degree to which the added fat reduced the loaf volume of bread baked from ether-extracted flours. However, different flours and a different baking test were used in the two cases and in addition the baking tests were conducted by different operators. Also there is considerable disagreement in the literature as regards the effect on loaf volume of adding fats in different proportions to the dough. Thus Herman and Hart (1927) found that the addition of 1 to 2% of a shortening agent represented as a "hydrostearoleine" did not affect the loaf volume, but the addition of 3% of the fat markedly reduced it. The results reported in this paper are therefore in accordance with those of Herman and Hart. Mohs (1915), on the other hand, found that the addition of various fats even to the extent of 10 to 15% operated to increase the loaf volume.

The effect of the added lard on the texture and color scores of the bread made from ether-extracted flour was to reduce them, but not to the extent that the improvement in these properties, due to ether extraction, was eliminated. The fact that the addition of lard reduced both color and texture scores is in agreement with the work of Treloar, Sherwood, and Bailey (1931) who found a significant positive correlation between color and texture.

On the basis of the effect of added fat on the gas-retaining ability of the dough it would appear that a reduction of loaf volume is to be expected. The data in Table II show that the volume attained by

TABLE III  
INCREASE IN VOLUME OF THE DOUGH AND INCREASE IN VOLUME OF DOUGH PLUS VOLUME OF CO<sub>2</sub> LOST FROM THE DOUGH FOR NATURAL  
AND ETHER-EXTRACTED STRAIGHT, 1ST CLEAR AND 2D CLEAR FLOURS

Time (min.)	Straight (natural)		Straight ether-extracted		1st Clear (natural)		1st Clear ether-extracted		2d Clear (natural)		2d Clear ether-extracted	
	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.	Increase in volume of the dough plus volume of the CO <sub>2</sub> lost from the dough	cc.
10	0	0	0	0	0	0	0	0	0	0	0	0
20	12	11	10	13	12	10	10	10	11	10	8	8
30	24	23	22	26	25	22	22	22	22	20	17	17
40	39	37	37	43	40	37	36	35	38	35	32	31
50	55	53	53	61	57	51	53	49	52	49	44	43
60	71	68	69	76	68	66	65	63	68	63	59	57
70	87	83	84	95	76	82	85	75	84	75	74	70
80	104	99	102	113	78	99	95	103	103	91	91	87
90	124	105	120	132	86	117	112	118	112	106	106	100
100	145	100	137	151	93	134	128	137	137	123	123	116
110	160	105	156	167	99	148	148	155	141	140	140	130
120	176	102	170	189	106	168	159	175	181	157	157	142
130	194	106	187	208	110	185	174	198	198	178	178	145
140	212	108	205	229	113	205	189	217	217	196	196	148
150	233	117	224	251	116	224	205	240	240	206	206	144
160	247	116	238	267	121	239	215	261	261	226	226	144
170	267	123	257	286	117	257	226	281	281	245	245	140
180	284	123	273	305	120	273	231	306	306	264	264	139
190	301	132	291	322	122	289	234	326	326	282	282	137
200	317	131	307	338	119	306	239	350	350	302	302	134
210	334	138	322	363	123	325	239	370	370	318	318	136
220	349	138	335	386	118	343	238	392	392	336	336	133
230	367	135	354	403	124	360	239	417	417	356	356	136
240	382	135	367	427	121	381	241	437	437	374	374	133

the dough prepared from ether-extracted flour was 343 cc. while the volumes of similar doughs containing 3 and 5% of added fat were 180 and 142 cc. respectively. It is therefore apparent that the addition of the lard markedly reduces the volume to which the dough is able to rise.

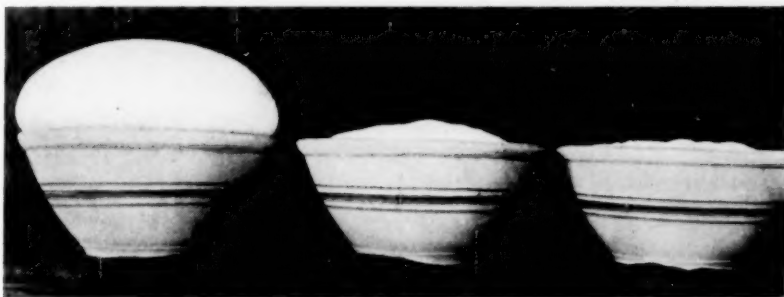


Fig. 2. The dough at the left was prepared from an ether-extracted flour, dough at the center from the natural flour and dough at the right from an ether-extracted flour containing 3% of added lard. All doughs were prepared from equivalent quantities of 3d middling flour and the fermentation time was 3 hours in every case.

In order to show more readily the effect of ether extraction and of addition of fat to the extracted flour on the volume to which the dough could rise, Figure 2 has been included. The dough at the left was prepared from ether-extracted flour, the second from natural flour, and the dough at the right from ether-extracted flour to which 3% of lard had been added.

Doughs of the same size were prepared and then allowed to ferment for three hours at the end of which time the doughs were photographed. The photograph indicates very clearly the effects of the various treatments accorded the doughs on the volumes to which they could rise.

### Discussion

In order to determine the mechanism by which bread of better color, texture and loaf volume was produced from ether-extracted and from natural flours a series of flours consisting of a 3d middlings, a straight, a 1st clear and a 2d clear flour were extracted with ether and the natural and ether-extracted flours subjected to tests in the Bailey-Johnson apparatus. Baking tests on these flours confirmed earlier work, viz., that bread produced from the ether-extracted flours was superior in volume, color and texture to that produced from natural flours.

Ether-extracted flours yielded doughs, the gas-retaining powers of which were remarkably superior to those of the corresponding natural

flours. Since the addition of fat reduced the gas-retaining power of the dough it appears that the fat increases the permeability of the dough surface so that the  $\text{CO}_2$  produced during fermentation readily escapes or that the presence of fat in the dough or vesicular surfaces reduces the extensibility of such surfaces so that they rupture before becoming extended to the same degree as is possible in doughs not containing ether soluble substances. It is most likely, however, that the ether soluble substances reduce the extensibility of the dough membrane rather than increase its permeability to carbon dioxide. An increased extensibility of the dough membrane would account for both increased volume and finer grained texture of the bread. Increased extensibility of the dough membrane due to ether extraction of the flour used in preparing the dough is therefore most probably the property which makes it possible to prepare bread of superior volume, color, and texture from ether-extracted flours.

### Conclusions

Bread of better color, volume and texture was produced from ether-extracted flour than from natural flour.

Doughs prepared from ether-extracted flours were markedly superior to natural flour doughs in their ability to retain the carbon dioxide produced during fermentation.

Addition of fat to doughs prepared from ether-extracted flours reduced their gas retaining powers.

Allowing the dough to rise several times with "punching" at the proper time resulted in decreasing the gas-retaining power of the dough.

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## THE COOKING OF CEREAL PORRIDGES

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(Read at the Convention, May, 1931)

When is a cereal porridge cooked? How much heating is necessary to cook it? The answers to these two questions were found to be very difficult to state definitely. It was found that no technical information was at hand which first of all defined the state of being cooked and secondly showed what was necessary to obtain that indefinite state. Definitions of the word "cook" indicate the action of heat; but the intensity of heat, its duration and its results are not considered.

In dealing with cereal porridges we have a raw product whose analysis indicates a large predominance of starch. The carbohydrate content, which runs about 65% to 70%, is composed of a very high percentage of starch and a small amount of such substances as sugars and the like. Of the carbohydrate other than starch, it may be said that their reactions with boiling water are of quite modest proportions when their respective quantities are in such minority as compared with the starch present. The action that takes place when starch and boiling water are in contact is covered by the broad term, gelatinization. A discussion of this questionable reaction is out of place in this paper but the end products of such a reaction form the visible physical characteristics of a cereal porridge that has been "cooked." The deduction may be drawn that a cereal porridge is "cooked" when 100% of its starch has been gelatinized. Thus the term "cooking" of a cereal porridge may be defined as the process of gelatinizing the starch. This stipulates that the degree of cooking is simply the percentage of gelatinization.

It is common knowledge that gelatinized starch in the presence of free iodine produces a color. Experience has shown that the color intensity of a starch-iodine mixture is dependent upon the amount

of starch present. Also a clear solution color is not produced by un-boiled starch, the color produced being carried in the starch particles and not dispersed throughout the solution. These properties of starch form the basis of a method which was devised to measure the amount of starch which had reacted with water. The results of such an analysis were then taken as the degrees of cooking.

### Method of Determining Gelatinized Starch in a Cereal Porridge

Choose the proper proportions of cereal, water and salt. First dissolve the salt in the water and bring to a vigorous boil on a hot gas flame. Add the cereal with constant stirring, which should require not over five seconds time. When vigorous boiling has again begun, transfer the beaker from the gas to a hot plate which previously has been adjusted so that it will keep the porridge just boiling. Keep the porridge covered with a watch glass to hold evaporation to a minimum.

At the end of the required time of boiling remove a large sample to a watch glass and cool as quickly as possible. From this sample weigh out, to the nearest .01 gram, a portion which is equivalent to 1 gram of the original dry cereal. This is accomplished by the use of the following formula:

$$\frac{\text{grams of cereal plus cc. of water plus grams salt}}{\text{grams of cereal}} =$$

grams of porridge which should be weighed out to be equivalent to 1 gram of dry cereal.

After weighing out the desired amount of porridge, place in an agate mortar with some tap water and rub it to a smooth suspension. Continue dilution until the mortar is full, let settle a minute or so and decant off the floating part of the porridge into a 1000 cc. volumetric flask. Repeat the grinding and floating operation until all the sample is washed into the flask, dilute to the mark with tap water and mix thoroughly.

The solutions necessary are (1), a 0.1% gelatinized starch solution, made by boiling 1 gram of pure starch in distilled water and then diluting to 1000 cc. and (2), an iodine solution, made up by diluting 5 cc. of a 0.5% tincture of iodine solution in distilled water to 1000 cc. with distilled water.

With these solutions the test is made by pipetting 5 cc. of the diluted sample solution, 40 cc. of tap water and 5 cc. of iodine solution, into a Nessler's tube. A series of standard tubes using known amounts of starch standard are next made up using the same amount of iodine and

water in order to produce a volume of 50 cc. The color of the test sample is matched with that of the proper standard and the amount of starch in the standard noted. A typical example of the test procedure follows.

30 grams of cereal, 200 cc. of water, and 1 gram of salt were boiled for 2 minutes, as described.

By use of the formula just given, the equivalent of 1 gram of dry cereal was found to be 7.78 grams.

$$\frac{30 \text{ plus } 200 \text{ plus } 1}{30} = 7.78 \text{ grams, the porridge equivalent of 1 gram of dry cereal.}$$

After trituration and dilution to 1000 cc., 5 cc. of the sample solution was found to be equal in color intensity to 2.5 cc. of standard starch solution. 5 cc. of the sample solution contains 5 milligrams of original cereal. 1 cc. of standard starch solution contains 1 mg. of starch. 2.5 cc. will contain 2.5 mgs. of starch. By proportion, therefore, the cc. of the sample is to the cc. of standard solution as 100 is to  $X$ . i.e.,  $5:2.5::100:X$ . By calculation  $X$  becomes 50, and represents the percentage of the cereal which has been gelatinized or cooked. This figure may in turn be re-calculated to represent the percentage of starch in the cereal by simply dividing the percentage of cereal gelatinized by the per cent of starch in the cereal, and multiplying by 100.

To avoid error, the following should be observed: (1) avoid soluble proteins; (2) keep the temperature of the color solution below 80° F. as higher temperatures dissipate the color; (3) standard starch solutions should not be used that are more than 3 to 4 hours old, as changes (probably hydrolysis) occurs in the starch which affects the color producing powers; (4) speed is often an important factor in handling the boiling operations; (5) distilled water is not necessary under ordinary conditions.

### Experimental

The experiments carried out by use of the above method were designed to give a picture of the cooking process of a cereal. The product used corresponds to an unpurified hard wheat middling. The following was the schedule of individual cookings:

- a. 30 gms. cereal, 200 cc. water or ratio 1-4.6 by volume.
- b. 30 gms. cereal, 220 cc. water or ratio 1-5.0 by volume
- c. 30 gms. cereal, 265 cc. water or ratio 1-6.0 by volume.
- d. 30 gms. cereal, 300 cc. water or ratio 1-7.0 by volume.

It should be stated that the ratios of cereal to water are stated in

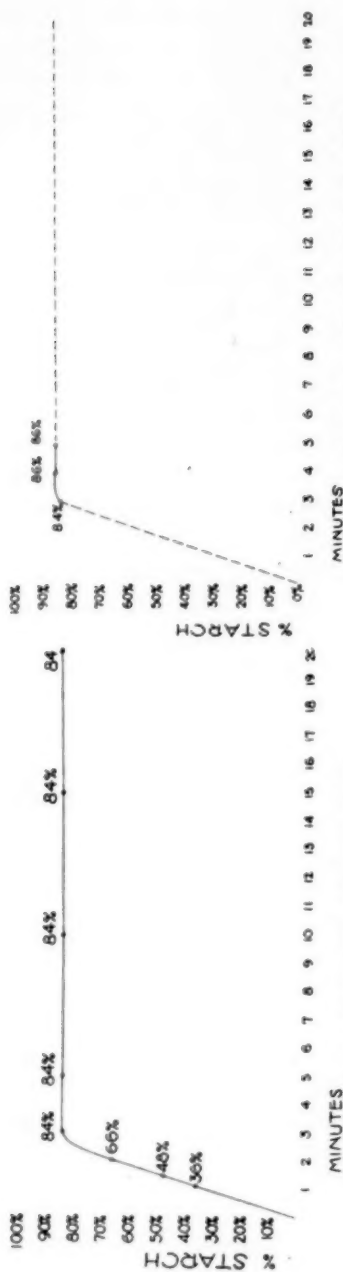


Fig. 1. Ratio 1-4.6; maximum gelatinization 84%.

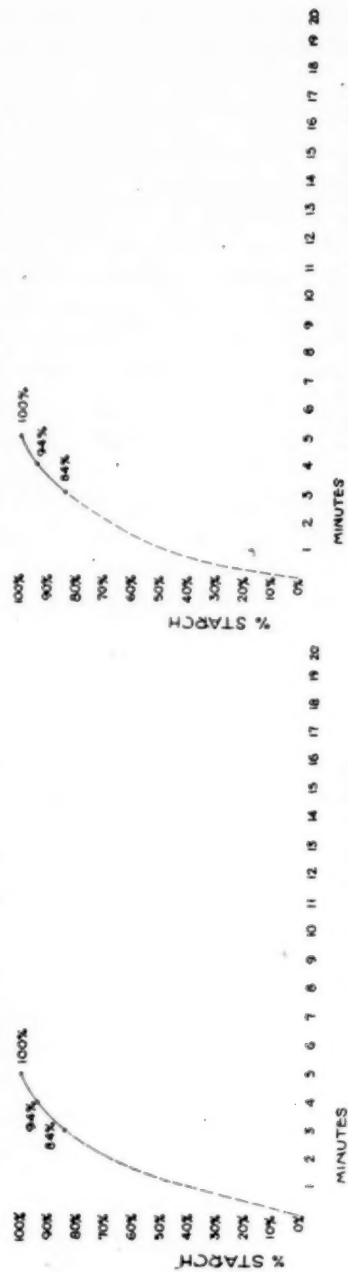


Fig. 2. Ratio 1-5.0; maximum gelatinization 86%.

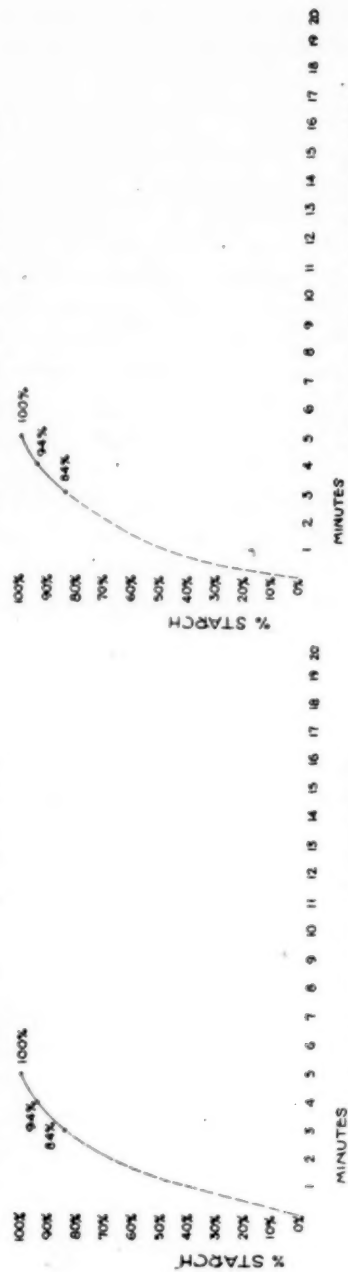


Fig. 3. Ratio 1-6.0; maximum gelatinization 100%.

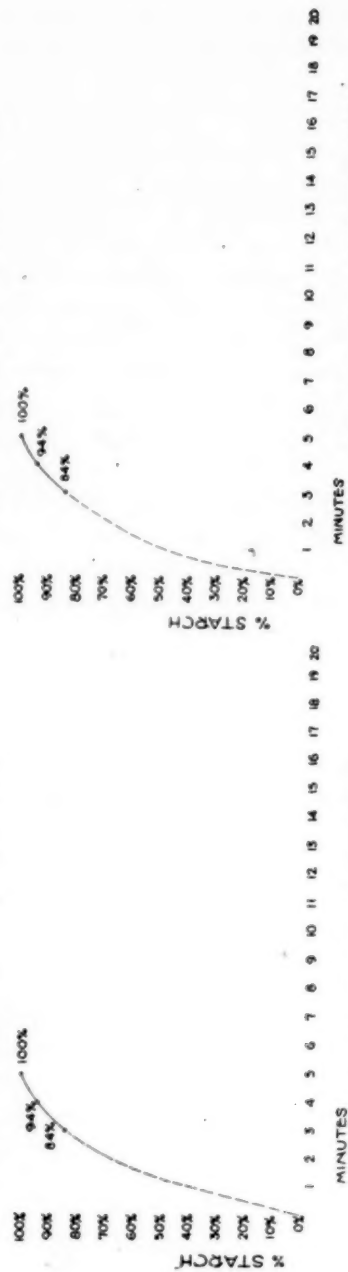


Fig. 4. Ratio 1-7.0; maximum gelatinization 100%.

Figures 1-4. Rate of Gelatinization as Influenced by Ratio of Cereal to Water.

terms of volume as well as weights so that comparisons with ordinary recipes could be easily made.

The cooking operations were timed as follows:

Group (a) 1, 1½, 2, 3, 5, 10, 15, 20 minutes.

Group (b) 3, 4, 5 minutes.

Group (c) 3, 4, 5 minutes.

Group (d) 3, 4, 5 minutes.

All tests such as 30 grams of cereal, 200 cc. of water, boiled for 5 minutes, were run in duplicate or triplicate and the results averaged. The data were plotted and appear in Figures 1, 2, 3, and 4.

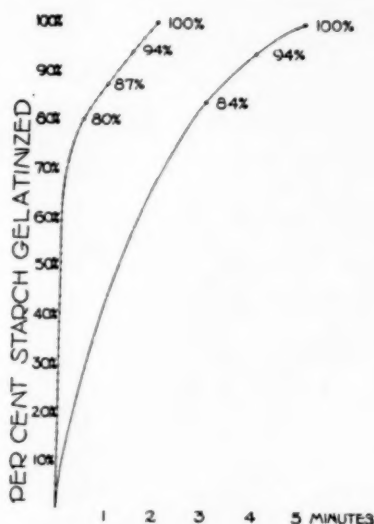


Fig. 5. Rate of Gelatinization as Influenced by Fineness of Division of Sample. Heavy line indicates coarsely ground material. Broken line indicates finely ground material. See text for mechanical analysis of samples.

The reason for not following out the full time schedule with all four groups can be seen from the graphs. In the cooking of Group *a*, as well as Group *b*, no change in gelatinization took place between 4 and 5 minutes as will be noted in Figures 1 and 2. Similarly, in the cooking of Groups *c* and *d*, gelatinization of the starch was complete before five minutes of cooking had elapsed.

The question arose as to the time of cooking required by a cereal which is more finely ground. Figure 5 gives a comparison of the cooking curve produced by treating a finely ground cereal in the same manner as the coarse product. The curve for the coarse product is the same as illustrated in Figure 3.

The mechanical analyses of the two products are as follows:

	Fine	Coarse
	Pct.	Pct.
Retained on a 20 mesh screen	0	21.9
Pass a 20 mesh, retained on 28 mesh	2.1	25.4
Pass a 28 mesh, retained on 32 mesh	23.4	30.0
Pass a 32 mesh, retained on IXX cloth	44.3	12.6
Pass a IXX cloth	30.2	10.1
	100	100

Figure 5 shows a very rapid rise in the gelatinized starch in the finer product. The greatest difficulty is that when 80% of the starch is transformed in 30 seconds time with the two unavoidable factors of narrow time limit and natural tendency for a porridge to retain a temperature above the reacting temperatures, error is liable to be great. Without much doubt the shape of this curve is somewhat different than a curve produced under ideal conditions. The only way that this could be done would be by freezing the reaction and means for this were not at hand.

### Conclusion

1. The term "cooking" as applied to cereals may be stated as "the process of gelatinizing the starch of a cereal."

2. A method of analysis of cereals for gelatinized starch is described. The method depends upon the color producing powers of gelatinized starch in an excess of iodine. By this method of analysis the gelatinization or cooking process can be traced.

3. The analysis of a cooked cereal porridge shows: (a) When sufficient water is present, cooking proceeds to completion at a rate which is a function of the granulation of the dry cereal; (b) When insufficient water is present, cooking proceeds at the normal rate for that cereal to a point at which the free water is apparently consumed. At this point gelatinization stops and further cooking is useless.



# A NOTE ON THE UTILITY OF THE TAG-HEPPENSTALL MOISTURE METER FOR DETERMINING THE MOISTURE CONTENT OF GROUND WHEAT <sup>1</sup>

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(Received for publication, May 15, 1931)

## Introduction

In the cereal laboratory the determination of moisture is an essential part of the laboratory routine in comparative studies of the protein and ash content of different wheats in order that the analytical results may be expressed on a uniform moisture basis. It has been the recent practice in this laboratory to run single determinations by the official vacuum oven method, since previous experience had shown that duplicate determinations rarely failed to agree within 0.3%, which was considered sufficiently close agreement for the purpose of reducing protein and ash results to a constant moisture basis. While the adoption of this procedure greatly reduced the time and labor involved, the capacity of a single analyst is approximately 100 determinations per day and moisture determinations still constituted a very laborious and time consuming portion of the laboratory work. Two projects involving several thousand protein determinations were under way when it was learned that the Canadian Co-operative Wheat Producers, Limited, had purchased a Tag-Heppenstall moisture meter and, through the kindness of Alan McLeod, their chief chemist, the apparatus was loaned to this laboratory.

This apparatus is designed for use on whole grain and, as a large number of the samples had already been ground it was decided to ascertain whether it would prove satisfactory for moisture determinations on the ground samples. The results obtained were deemed of sufficient interest to warrant recording.

## Description of the Tag-Heppenstall Moisture Meter

The estimation of moisture by the Tag-Heppenstall moisture meter depends upon the variation in electrical resistivity with changing moisture content of grain being crushed between revolving corrugated roll electrodes. The measurement of the resistance is based on Ohm's Law

<sup>1</sup> Published as Paper No. 22 of the Associate Committee on Grain Research, National Research Council of Canada.

<sup>2</sup> Wheat Pool Fellow for Manitoba, 1930, Canadian Co-operative Wheat Producers, Ltd., Winnipeg.

$I = \frac{E}{R}$ , the circuit being so designed that corrections for variations in  $E$  may be made by altering the resistance in the ammeter circuit. Under these conditions the strength of the current flowing is inversely proportional to the resistance and the ammeter readings may be interpreted as a measure of the moisture content of the sample by reference to a conversion chart.

Figure 1 is a diagrammatical representation of the circuit which

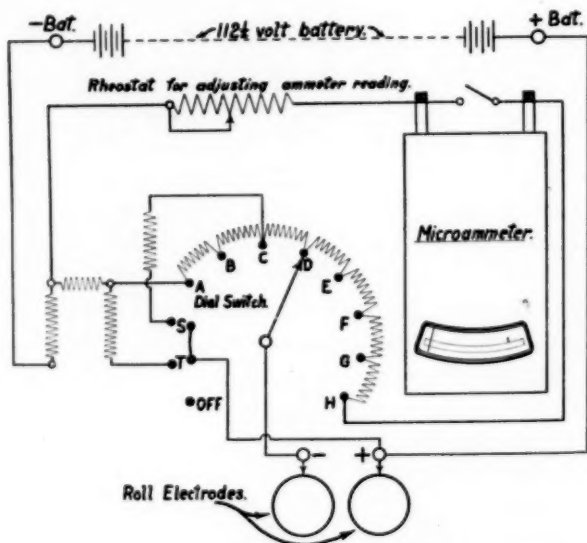


Fig. 1. Wiring diagram of Tag-Heppenstall moisture meter.

comprises a high voltage battery, a microammeter with an adjustable rheostat, an Ayrton shunt having eight contacts lettered *A* to *H*, with two extra contacts, *S* and *T*, which are connected to standard resistors for testing the batteries, roll electrodes, and a protective resistor in series with the batteries to prevent a short circuit from injuring the ammeter. The roll electrodes are finely corrugated, the distance between them being adjustable, and are driven by bakelite gears, the whole being enclosed in a bakelite container provided with a feed hopper.

Prior to making a determination, the ammeter deflection is first standardized by turning the dial switch to *S* and adjusting the rheostat so that the meter pointer stands at 50 on the scale. In order to avoid appreciable error from internal resistance of run down batteries, the test point *T* is provided. When the tap *T* is used more current is drawn from the batteries than when *S* is employed. Run down

batteries are thus indicated by a low reading of the ammeter when testing on *T* after standardizing on *S*.

If then a reading of 50 is obtained on passing a sample of grain through the roll electrodes with the dial on *C*, the resistance of the sample is equal to that of the standard resistor connected to *S*. Thus, depending on the position of the dial switch and the deflection of the ammeter, the ratio of the resistance of the sample to that of the standard is ascertained.

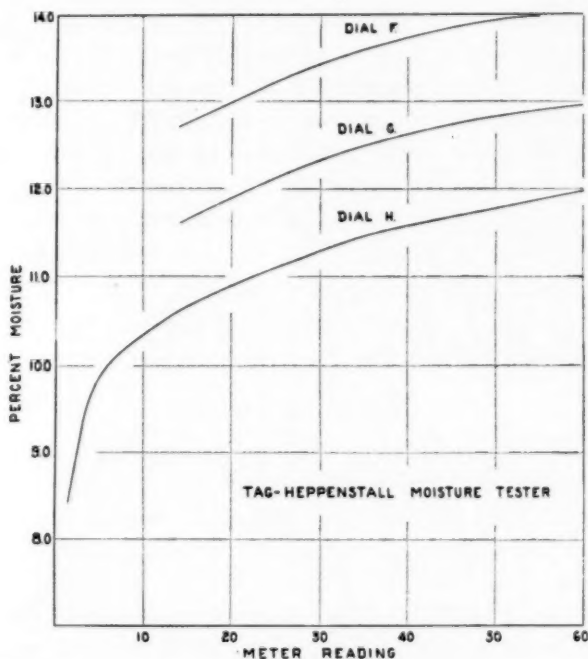


Fig. 2. Typical curves showing the relation between microammeter deflection and moisture content of hard red spring wheat.

In determining the moisture content of whole wheat the distance between the roll electrodes is adjusted to .040 inch which is designated as 40 on the scale provided. About 50 grams of wheat are placed in the hopper and the handle given a partial turn to bring some of the grain between the rolls. The dial switch is then set on the appropriate contact to give a deflection of between 15 and 60 on the ammeter, the handle of the roll electrodes turned at a rate of about 120 r.p.m., and the average deflection read. Since the resistance of the grain decreases with increasing moisture content the fraction of the current flowing through the ammeter circuit must be decreased to secure a suitable deflection. This is accomplished by moving the dial setting to tap

*G, F, E*, etc., with the increasing moisture content of the samples being tested.

Conversion charts for hard red winter, hard red spring, durum, and soft red winter wheats, giving the percentage moisture corresponding to various ammeter deflections and dial settings, are provided with the instrument, covering a range in moisture of 8.2% to 23.8%.

Figure 2 illustrates the relation between moisture content and ammeter deflection for a few of the dial settings. It will be observed that the curve for setting *H* rises sharply in the region of 8% to 10% moisture. For example, with wheat in the region of 8.3% moisture an increase in moisture content of 0.1% is indicated by an increased deflection of only 0.2, whereas, in the region of 11.5%, a similar variation in moisture content results in an increased deflection of 5.

Temperature is an important factor in manipulating the instrument since electrical resistance of grain decreases with increasing temperature. The conversion charts supplied are compiled for a wheat temperature of 25° C. or 77° F. The temperature correction given with the instrument is 0.04% moisture for 1° F. deviation from 77° F., the correction being additive for temperatures lower than the calibration temperature.

### Experimental

A few preliminary trials on hard red spring wheat ground so that at least 50% passed through a 36 grits gauze sieve revealed that such material would feed through the rolls uniformly and give a steady reading when the distance between the rolls was set at .020 inch—the closest adjustment available. The ammeter deflections when converted to moisture, using the conversion chart for whole wheat, agreed closely with the values obtained by the official vacuum oven method. Accordingly, comparative data on the moisture content of 335 ground wheat samples were collected, single determinations being run by each method. Two days prior to making the determinations with the Heppenstall apparatus the ground samples were placed in the same room as the moisture meter, and the readings taken at a temperature of 22° C. The results are recorded in Table I below, as the frequency distribution of the differences between the moistures obtained by the two methods and calculated by subtracting the vacuum oven values from the results obtained by the Tag-Heppenstall moisture meter.

TABLE I  
FREQUENCY DISTRIBUTION OF DIFFERENCES BETWEEN MOISTURE RESULTS

Difference	Frequency	Frequency in per cent
P.ct.		
-.5	3	0.9
-.4	8	2.4
-.3	26	7.8
-.2	33	9.9
-.1	42	12.5
0	54	16.1
+.1	56	16.7
+.2	56	16.7
+.3	37	11.0
+.4	13	3.9
+.5	5	1.5
+.6	1	0.3
+.7	1	0.3
Total	335	100.0

Summarizing the differences recorded in Table I, 45.4% of the samples analyzed gave an agreement by the two moisture methods within  $\pm 0.1\%$  moisture; 72.0% within  $\pm 0.2\%$ , and 90.8% within  $\pm 0.3\%$ .

The range in moisture content and the statistical constants calculated from the data are given in Table II.

TABLE II  
RANGE IN MOISTURE AND STATISTICAL CONSTANTS CALCULATED FROM MOISTURE DATA

	Moisture method	
	Tag-Heppenstall	Vacuum oven
Range in moisture content of samples	8.5-12.5%	8.5-12.5%
Mean moisture in per cent	11.007	10.966
Mean difference (T.H. method—vacuum oven method)		0.041%
Standard deviation of differences		0.218%
Probable error of mean difference		$\pm 0.008\%$
Correlation coefficient (T.H. method versus Vacuum oven method)	$+0.924 \pm 0.005$	

A comparison of the mean difference in the moisture results by the two methods with its probable error reveals that it is significant. However, the difference only amounts to 0.041% and can hardly be regarded of any practical importance. In view of the fact that single moisture determinations were run by the vacuum oven method, and

that 90.8% of the samples analyzed gave an agreement within  $\pm 0.3\%$  moisture by the two methods, the Tag-Heppenstall moisture meter may be regarded as giving satisfactory results on ground wheat using the same conversion chart as for whole wheat.

On the basis of these results the machine has been employed in this laboratory for the purpose of securing moisture data to be used in reducing protein and ash results to a uniform moisture basis. In the grinding of wheat there is frequently a considerable and variable loss of moisture, especially when the samples are put through the grinder in rapid succession, resulting in heating of the grinding plates, or when the moisture content exceeds about 13.0% at the ordinary relative humidity prevailing in this laboratory. Under these conditions the moisture content of the whole wheat is liable to vary appreciably from that of the ground sample on which the chemical determinations are made, and the use of the Tag-Heppenstall moisture meter directly on the ground sample greatly increases its utility in the cereal research laboratory. The machine as at present constructed was not found satisfactory for use on flours, as they did not feed uniformly through the rolls, giving a fluctuating deflection of the ammeter. In conclusion, the results reported here are not to be regarded as a critical comparative study of the Tag-Heppenstall moisture meter with the vacuum oven method, as this would involve much more extensive work over a wider range in moisture content.

#### Acknowledgments

The authors wish to acknowledge the financial assistance of the Canadian Co-operative Wheat Producers, Limited, Winnipeg, and their kindness in loaning the moisture meter. They are also indebted to the C. J. Tagliabue Company, Brooklyn, New York, for the description and wiring diagram of the apparatus, and to L. D. Sibbitt and D. S. Binnington for technical assistance.



# THE COMPOSITION OF BREAD

C. B. MORISON

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(Read at the Convention, May, 1931)

The American Institute of Baking has recently made analyses of bread from representative bakeries located in different parts of the country. The bread was weighed at the bakery one hour after baking, wrapped, packed securely and consigned to the Institute at Chicago.

On arrival at the laboratory, the samples of bread were weighed, sliced, dried, and ground for the determination of moisture, protein ( $N \times 6.25$ ), crude fiber, fat or crude ether extract, and ash. The methods used were those of the Association of Official Agricultural Chemists as published in their manual, "Official and Tentative Methods of Analysis." and the Journal of the Association of Official Agricultural Chemists.

TABLE I

ANALYSIS OF BREAD—MOISTURE CONTENT ONE HOUR AFTER BAKING

Sample number	Water	Solids	Protein ( $N \times 6.25$ )	Nitrogen-free extract	Crude fiber	Fat	Ash	Remarks
	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	
11	37.63	62.37	9.90	47.61	0.31	2.65	1.90	
13	36.54	63.46	9.96	48.54	0.28	2.75	1.93	
15	37.13	62.87	9.55	48.22	0.29	3.11	1.70	
28	37.58	62.42	9.64	47.68	0.25	2.86	1.99	
34	36.63	63.37	9.79	48.67	0.27	2.89	1.75	
36	37.50	62.50	9.99	47.28	0.15	3.01	2.07	Sliced bread
38	36.91	63.09	9.47	49.00	0.25	2.69	1.68	
44	35.84	64.16	9.50	49.60	0.27	3.13	1.66	
46	37.68	62.32	9.79	47.12	0.36	3.18	1.87	
48	36.93	63.07	9.86	48.36	0.28	2.73	1.84	
75	32.76	67.24	9.45	52.59	0.35	3.00	1.85	
77	37.37	62.63	9.23	48.43	0.33	2.85	1.79	
201	36.81	63.19	9.52	48.61	0.30	3.03	1.73	
203	37.43	62.57	9.35	47.85	0.17	3.50	1.70	
205	37.26	62.74	9.39	48.24	0.38	3.02	1.71	
221	37.57	62.43	8.86	48.65	0.33	3.02	1.57	
225	36.12	63.88	9.21	49.42	0.49	3.01	1.75	
229	37.52	62.48	8.39	48.46	0.40	3.50	1.73	
246	36.04	63.96	9.30	49.38	0.53	3.12	1.63	
248	36.76	63.24	9.34	48.74	0.47	3.02	1.67	
254	37.79	62.21	8.97	47.35	0.88	3.22	1.79	
333	38.00	62.00	9.08	47.84	0.44	2.91	1.73	Sliced bread
335	37.29	62.71	10.26	47.09	0.55	2.98	1.83	
346	37.47	62.53	9.17	48.78	0.21	2.73	1.64	
337	35.87	64.13	9.02	50.12	0.39	2.77	1.83	
Average	36.90	63.10	9.44	48.54	0.36	2.99	1.77	
Max.	38.00	62.00	10.26	52.59	0.88	3.50	2.07	
Min.	32.76	67.24	8.39	47.09	0.15	2.65	1.57	

The samples of bread sent for analysis included whole wheat, rye, raisin, salt rising, and potato, but the majority of the samples was white bread made with milk, both sliced and unsliced.

The results of the analysis of twenty-five different samples of white bread have been collected and summarized in Tables I and II. Table I

TABLE II  
ANALYSIS OF BREAD CALCULATED TO DRY BASIS

Sample number	Protein (N×6.25)	Nitrogen-free extract	Crude fiber	Fat	Ash
	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.
11	15.87	76.33	0.50	4.25	3.05
13	15.69	76.50	0.44	4.33	3.04
15	15.19	76.70	0.46	4.95	2.70
28	15.44	76.39	0.40	4.58	3.19
34	15.45	76.80	0.43	4.56	2.76
36	15.98	75.65	0.24	4.82	3.31
38	15.01	77.67	0.40	4.26	2.66
44	14.81	77.30	0.42	4.88	2.59
46	15.71	75.61	0.58	5.10	3.00
48	15.63	76.68	0.44	4.33	2.92
75	14.05	78.22	0.52	4.46	2.75
77	14.74	77.32	0.53	4.55	2.86
201	15.06	76.93	0.47	4.80	2.74
203	14.94	76.47	0.27	5.60	2.72
205	14.97	76.88	0.61	4.81	2.73
221	14.19	77.93	0.53	4.83	2.52
225	14.42	77.36	0.77	4.71	2.74
229	13.43	77.56	0.64	5.60	2.77
246	14.54	77.20	0.83	4.88	2.55
248	14.77	77.07	0.74	4.78	2.64
254	14.42	76.11	1.41	5.18	2.88
333	14.65	77.16	0.71	4.69	2.79
335	16.36	75.09	0.88	4.75	2.92
346	14.66	78.01	0.34	4.37	2.62
337	14.07	78.15	0.61	4.32	2.85
Average	14.96	76.92	0.57	4.74	2.81
Max.	16.36	78.22	1.41	5.60	3.31
Min.	13.43	75.09	0.24	4.25	2.52

summarizes the proximate analyses of the bread on the basis of the water and solids content of the bread one hour after baking, and in Table II the analytical results have been calculated to the dry basis for purposes of comparison.

It is of interest to compare the results of these analyses with others that previously have been published. The Connecticut Agricultural Experiment Station in 1913<sup>1</sup> published an extensive report of the analysis of "two hundred and one loaves, representing the product of seventy-nine Connecticut, one Springfield and three New York bakeries." The average composition of these samples of bread, calculated to the dry basis was as follows:

<sup>1</sup> J. B. Street, et al. Conn. Expt. Sta. Ann. Rpt. Part 4, pp. 257-266.

TABLE III  
AVERAGE COMPOSITION OF BREAD—DRY BASIS

Connecticut Agricultural Experiment Station, 1913		American Institute of Baking, 1931	
Protein (N×6.25)	14.69%	Protein (N×6.25)	14.96%
Carbohydrates (N.F.E.) including crude fiber	81.33%	Carbohydrates (N.F.E.)	76.92
Ether extract (Fat)	1.83%	Crude fiber	0.57
Ash	2.15%	Ether extract (Fat)	4.74
		Ash	2.81
	100.00%		100.00%

The slight increase in the protein, high increase in fat, and the considerably higher ash content with decrease in total carbohydrates in the samples of bread recently analyzed here are of considerable interest. The high increase in fat content reported is due in part to the use of the modified method, which always gives higher and more consistent results with such material as bread than the older direct ether extraction, but it also probably indicates the comparatively recent tendency of the baking industry to use larger amounts of shortening in the bread than formerly. The increase in protein may be ascribed to the general practice of using larger quantities of milk than previously, and the increase in ash partially may be attributed to the same ingredient. It is not possible at this time to report the total sodium chloride content of the ash, the sodium chloride free ash and the total percentage of calcium and phosphorous respectively.

It may not be out of place to point out here, that most textbooks and general works on food and nutrition are still publishing analyses of bread made many years ago, based on the compilations of Atwater and Bryant which now are out of date. A widely known authority (Sherman)<sup>2</sup> states the composition of white bread to be as follows:

*Average of 198 Samples.*—Water, 35.3%; protein (N×6.25), 9.2%; total carbohydrates including fiber, 53.1% (fiber 0.5%, av. 27 analyses); fat, 1.3%; ash, 1.1%; total, 100.0%.

Such analytical data has historical value, but it does not give the student authentic information of the average composition of bread as produced today according to recent analyses, which indicate a somewhat higher protein, fat, and ash with a decrease in total carbohydrates as might be expected from a more general use of larger amounts of milk and shortening.

<sup>2</sup> H. C. Sherman, Food Products, p. 326. The MacMillan Co., New York.

## A NOTE ON THE WEIGHT OF A THOUSAND KERNELS

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(Received for publication, April 6, 1931)

The test weight per bushel, or the weight in pounds of a bushel of wheat, is a well-known indication of plumpness of grain, and is determined in most mills as a routine test. It is an important characteristic of wheat and is one of those taken into account in most grading systems. Thus, Nos. 1 and 2 Northern Manitoba wheats must, by Statute, weigh at least 60 pounds and 58 pounds per bushel, respectively; while No. 3 Northern Manitoba must weigh at least 57 pounds per bushel. In the United States of America, wheats grading No. 1, of all classes, must weigh at least 60 pounds,<sup>1</sup> and grade No. 2, of all classes, must weigh at least 58 pound per bushel.<sup>2</sup> Plate wheats (Argentine) are also sold on the basis of a stated bushel weight, or hectolitre weight (i.e., the weight in kilograms of a hectolitre of the wheat).

In some cereal laboratories, e.g. those concerned with plant breeding experiments, actual size of grain is of interest quite apart from the fact that the supplies of grain available are often too small for the bushel weight to be determined. In such cases the weight of 1000 kernels (sometimes even of 100) is determined.

In general, a close correlation between plumpness, as measured by test weight per bushel, and weight per 1000 kernels is not to be expected and probably would not be found except between different grades of the same wheat, e.g. Nos. 1 to 6 Northern Manitoba. Plumpness or high bushel weight is associated with high flour yield which is the aspect of the problem of most interest to millers. On the other hand these two characters are not necessarily and directly influenced by size of grain. Thus large berried Australian wheat does not necessarily yield more flour than the smaller berried Manitoba wheat, although the differences in weight per 1000 kernels of the two wheats are great.

A search of the literature has not revealed any investigation in which the reliability of the determination of the weight per 1000 kernels has been established. This is surprising in view of the well known variability in size and plumpness of grain, even from the same ear of wheat.

<sup>1</sup> It should be remembered in this connection, that in the U.S.A., the American or Winchester, and not the Imperial bushel is used. The Imperial bushel = 2,218.192 cu. ins.; the American bushel = 2,150.420 cu. ins. Hence one Imperial bushel = 1.03222 American bushels. E.g. 60 lb. per American bushel = 61.9 lb. Imperial bushel.

<sup>2</sup> Except the Hard Red Spring wheats of the sub-classes Dark Northern Spring, Northern Spring, and Red Spring, of which the bushel weights are 58 lb. and 57 lb. respectively.

Two types of wheat, No. 1 Northern Manitoba and Hard Federation (Pacific), have been examined in these laboratories in order to test the accuracy and range of variation of the weight of 1000 kernels. No. 1 Northern Manitoba must contain, by Statute, sixty-five per cent. of hard red vitreous kernels not necessarily of the same variety, and the grade may include all varieties of hard red spring wheat grown in Canada and equal in value to Marquis wheat. It is therefore a mixture of commercially similar wheats. Hard Federation wheat on the other hand, although containing some admixture, is a reasonably homogeneous commercial variety. Each sample was thoroughly mixed and spread out on the laboratory bench. By means of a small spatula 1000 kernels were counted out, placed in a weighed container and weighed to the nearest milligram. No deliberate selection of seeds was made, the operator taking the seeds nearest to hand. All broken and obviously damaged grains were rejected but no whole undamaged kernels were rejected merely on account of unusual size, i.e. either very large or very small.

Altogether, ninety, separate thousands of the No. 1 Northern Manitoba wheat and seventy, separate thousands, of the Hard Federation wheat were counted and weighed. The weights of 1000 No. 1 Northern Manitoba kernels varied from 31.61 to 38.66 grams with a mean value of 34.47 grams; and 1000 Hard Federation kernels varied from 27.93 to 33.34 grams, with a mean value of 30.42 grams.

In Table I are given the weights arranged in ascending order at group intervals of 0.5 gram, and the corresponding frequencies. It will be noticed that when the weights are arranged in one way, e.g., with groupings 32.00 to 32.49, 32.50 to 32.99, etc., the frequencies are somewhat irregular, the corresponding frequency curves being bimodal. If, however, the weights are arranged slightly differently, e.g., 32.25 to 32.74 grams, 32.75 to 33.24 grams, etc., the frequencies are regularly, but slightly unsymmetrically distributed, the corresponding frequency curves being mono-modal, but slightly unsymmetrical. These differences may be due to the relatively small numbers of samples weighed or to variations in sampling. However, the dissymmetry of the frequency curves is slight, and the modes and the means do not differ by more than 0.25 gram so that in view of the fact that the weight of 1000 kernels is not a measurement of any great precision the calculation of approximate probable errors of these weights seems to be justified. The probable errors of the single determinations ( $PE_s$ ) were calculated by means of the formula:

$$PE_s = 0.6745 \sqrt{\frac{\sum d^2}{n-1}},$$

and the probable errors of the means ( $PE_m$ ) of the seventy and of the ninety thousands from the formula:

$$PE_m = 0.6745 \sqrt{\frac{\Sigma d^2}{n(n-1)}}$$

TABLE I

FREQUENCY DISTRIBUTIONS OF WEIGHTS PER 1000 KERNELS OF NO. 1 NORTHERN MANITOBA AND HARD FEDERATION (PACIFIC) WHEAT

<i>No. 1 Northern Manitoba</i>			
Grouping in Grams	Frequencies	Grouping in Grams	Frequencies
31.0 to 31.49	0	30.75 to 31.24	0
31.5 to 31.99	1	31.25 to 31.74	1
32.0 to 32.49	0	31.75 to 32.24	0
32.5 to 32.99	6	32.25 to 32.74	1
33.0 to 33.49	9	32.75 to 33.24	9
33.5 to 33.99	9	33.25 to 33.74	9
34.0 to 34.49	19	33.75 to 34.24	9
34.5 to 34.99	11	34.25 to 34.74	20
35.0 to 35.49	25	34.75 to 35.24	16
35.5 to 35.99	4	35.25 to 35.74	16
36.0 to 36.49	3	35.75 to 36.24	4
36.5 to 36.99	1	36.25 to 36.74	2
37.0 to 37.49	0	36.75 to 37.24	1
37.5 to 37.99	1	37.25 to 37.74	0
38.0 to 38.49	0	37.75 to 38.24	1
38.5 to 38.99	1	38.25 to 38.74	1
39.0 to 39.49	0	38.75 to 39.24	0
		Mode 34.62 gms.	
		Mean 34.47 gms.	
<i>Hard Federation</i>			
27.0 to 27.49	0	27.25 to 27.74	0
27.5 to 27.99	2	27.75 to 28.24	2
28.0 to 28.49	0	28.25 to 28.74	0
28.5 to 28.99	4	28.75 to 29.24	5
29.0 to 29.49	2	29.25 to 29.74	5
29.5 to 29.99	12	29.75 to 30.24	14
30.0 to 30.49	15	30.25 to 30.74	13
30.5 to 30.99	8	30.75 to 31.24	8
31.0 to 31.49	6	31.25 to 31.74	9
31.5 to 31.99	12	31.75 to 32.24	6
32.0 to 32.49	5	32.25 to 32.74	5
32.5 to 32.99	2	32.75 to 33.24	2
33.0 to 33.49	2	33.25 to 33.74	1
33.5 to 33.99	0	33.75 to 34.24	0
		Mode 30.20 gms.	
		Mean 30.42 gms.	

in which  $\Sigma d^2$  equals the sum of the squares of the deviations of the individual determinations from the mean of all determinations, and  $n$  equals the number of tests, i.e. the number of thousand kernels weighed.

The results are illuminating. The probable errors of the means



were  $\pm 0.08$  for the Manitoba set, and  $\pm 0.097$  for the Federation weights. That is the weight of 1000 No. 1 Northern Manitoba kernels (the mean of ninety replicate determinations) was  $34.47 \pm 0.08$  grams; the weight of 1000 Hard Federation kernels (the mean of seventy replicate determinations) was  $30.42 \pm 0.097$  grams. For two wheats to be significantly different in mean weight of 1000 kernels the mean weights would normally differ by more than three times the probable error of the difference, or in the two cases under consideration by more than 0.37 gram. (The probable error of the difference of two means,  $x$  and  $y$ , may be obtained from the  $P.E.$ 's of the means by the following formula:  $PE_{x-y} = \sqrt{PE_x^2 + PE_y^2}$ , or more simply and with sufficient accuracy, by multiplying the mean of the two  $P.E.$ 's by  $\sqrt{2}$ .) The two mean weights actually differ by more than four grams and there is, therefore, no doubt that the No. 1 Northern Manitoba wheat has a definitely higher weight per 1000 kernels by about 12 per cent., than the Federation wheat. In practice seventy to ninety replicate determinations are not made, but only one or two. The probable errors of *single* determinations of the weight of 1000 kernels of No. 1 Northern Manitoba and of Hard Federation were  $\pm 0.75$  and  $\pm 0.81$  respectively, and the  $PE$  of the difference of the mean weights was  $\pm 1.1$ . Three times the latter probable error equals 3.3, and for these two wheats as sampled to differ significantly in weight per 1000 kernels, *when only one determination is made on each*, the observed difference would be more than 3.3 grams. The actual observed difference of the mean values was 4.05 which would be significant if the mean values had been single values.<sup>3</sup> If the determinations are carried out in duplicate the probable error of the difference of the means works out at approximately  $\pm 0.70$  in which case the weights could not be regarded as significantly different unless they varied by more than  $0.70 \times 3$ , or 2.1 grams.

This range of variations was so considerable that errors due to sampling were suspected. The determinations were repeated on the same bulk of Hard Federation wheat using a somewhat different method of sampling. A glass tube, sealed at one end and of such a size that when full of wheat it contained approximately 1000 kernels, was used as a scoop. Ninety-eight such scoopfuls were taken from a thoroughly mixed bulk of wheat contained in a large tin. All the whole kernels contained in each scoopful were separated from the residue of broken grains, etc., counted and weighed, and the weight of 1000 kernels calculated from the known weight of  $x$  kernels;  $x$  varied between 675 and 1065.

<sup>3</sup> The two single values nearest the mean values were: No. 1 Northern Manitoba 34.476 gms., Hard Federation 30.406 gms.

In this series the weights of 1000 kernels varied from 26.9 to 31.9 grams with a mean value of 29.0 grams. As will be seen from Table II the frequency curve was symmetrical and mono-modal. The probable error of the mean was,  $PE_m = \pm 0.055$ , and that of a single determination was,  $PE_s = \pm 0.55$ . This is still high but is a marked improvement on the earlier values and indicates that sampling errors had been reduced by the new method of sampling adopted. From these

TABLE II  
FREQUENCY DISTRIBUTION OF WEIGHTS PER 1000 KERNELS OF HARD FEDERATION WHEAT.<sup>1</sup>

Grouping in Grams	Frequencies
26.0 to 26.49	0
26.5 to 26.99	1
27.0 to 27.49	2
27.5 to 27.99	4
28.0 to 28.49	15
28.5 to 28.99	26
29.0 to 29.49	25
29.5 to 29.99	11
30.0 to 30.49	10
30.5 to 30.99	3
31.0 to 31.49	0
31.5 to 31.99	1
32.0 to 32.49	0
Mode 29.0	
Mean 29.0 gms.	

<sup>1</sup> Different method of sampling from that used for data in Table I.

data it appears that the probable error of duplicate determinations would be  $\pm 0.39$ , and that of the difference of the means of duplicates of simpler  $PE \pm 0.55$ . On this basis, for two wheats (sampled in the manner described) to differ significantly in weight per 1000 grains the observed difference in weights should be greater than 1.65 grams.

### Summary

It is evident from the results discussed that the weight per 1000 kernels is an unreliable determination unless a particular method of sampling the grain is adopted and the probable error of the determination for that method of sampling is specified.

## CHEMICAL LEAVENING AGENTS AND THEIR CHARACTERISTIC ACTION IN DOUGHS

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Chemical leavening agents are best known to the consuming public as baking powders. Little attention has been paid, aside from that learned through practical experience, as to what happens to them in a dough or batter before the product is baked. In consequence, it was thought of sufficient importance to devise a method suitable for the study of the action of various acid components of baking powders under actual baking conditions. Investigations have already been reported in which the chemical reactions of baking powders have been discussed and attempts have been made to formulate equations to represent these reactions. These will not be discussed here. Smith and Bailey (1923) presented data showing the effect of baking powder residual salts upon the colloidal properties of bread doughs.

The decomposition of baking powders, and the evolution of carbon dioxide, has for the most part been investigated in water solution in the cold and conclusions drawn as to their relative efficiency. Halliday and Noble (1928) state that the expectations from such experiments appear to be confirmed by actual baking experiments. That this method does not study the baking powder under the actual conditions of reaction in doughs is at once evident. It is the purpose here to present information obtained under more practical conditions. Although other workers in this field may have had this in mind and possibly have recorded observations similar in nature, no publications have appeared in this connection other than that published recently by Noble and Halliday (1931). Their work was limited to a study of the percentage of carbon dioxide evolved after 40 seconds mixing of ingredients such as are used in making muffin batters and the percentage of carbon dioxide lost from the dough after 40 seconds mixing and after one hour standing of the batter. These authors worked with commercial baking powders of the tartrate, phosphate, and phosphate-sodium aluminum sulphate types.

In considering the problem of distribution of the carbon dioxide made available for baking purposes by the reaction of baking powder components three divisions are at once evident: 1. the amount lost from a dough during mixing and standing; 2. the amount causing expansion of the dough; this is called "bench action" by bakers; 3. the

amount of carbon dioxide dissolved or adsorbed and that available from unreacted soda which will be effective as leavening at oven heat.

The experimental determination of that part of the total gas which goes into each of these three divisions when different types of baking acids are used, has been attempted and is to be presented here. Results of baking tests correlating the gas evolution results are also given.

## Experimental

### MATERIALS USED

For the purposes outlined above four baking acids were selected as representative of the types found in common use in the home and bake-shop whether added as such in conjunction with soda or in the form of baking powder. The acids selected, calcium acid phosphate, sodium acid pyrophosphate, potassium acid tartrate (cream of tartar) and phosphate-sodium aluminum sulphate, were from commercial sources and were used at their commercial neutralizing values.

Two types of flour mixes, the biscuit type and the doughnut type were selected to represent commercial baked products in which chemical leavening agents are used for aerating purposes. The biscuit type was considered to be representative of pancakes, hot breads and biscuits, and, for experimental purposes, dry mixes included flour, soda 1.5% (flour basis), baking acid at the proper neutralizing strength, salt 1.75%, and shortening 10%, with water as the liquid. The doughnut type of mix was considered as representative of waffles, muffins, cakes and doughnuts, in which sugar and eggs are included in addition to the above ingredients and with milk in place of water. For convenience in mixing and handling, dry doughnut flour mixes were made up of flour, shortening 5.55%, sugar 22.2%, powdered egg 3.33%, dry skim milk 10%, and salt 1%. To aliquots of this mix, 1% of soda and a baking acid were added in the proper proportions and water was used as the liquid.

### DESCRIPTION OF APPARATUS AND METHOD OF OPERATOR

The measurement of the loss of leavening gas during the mixing of doughs was approached in this laboratory some three years ago by the use of a small enclosed bomb type mechanical mixer for measurement of the rate of reaction of baking powders in doughs. It was early recognized that two sources of error could be overcome by the construction of an efficient enclosed mixer and by temperature control. Figure 1 shows the construction of the apparatus that has been in use in this laboratory for over two years. The mixer *A* is of steel construction, copper plated to prevent rusting. A mixing arm extends inward

through a mercury seal and has two pins which rotate in concentric circles against stationary baffle pins extending upward from the bottom of the mixer. The mercury seal forms a portion of the lid which can

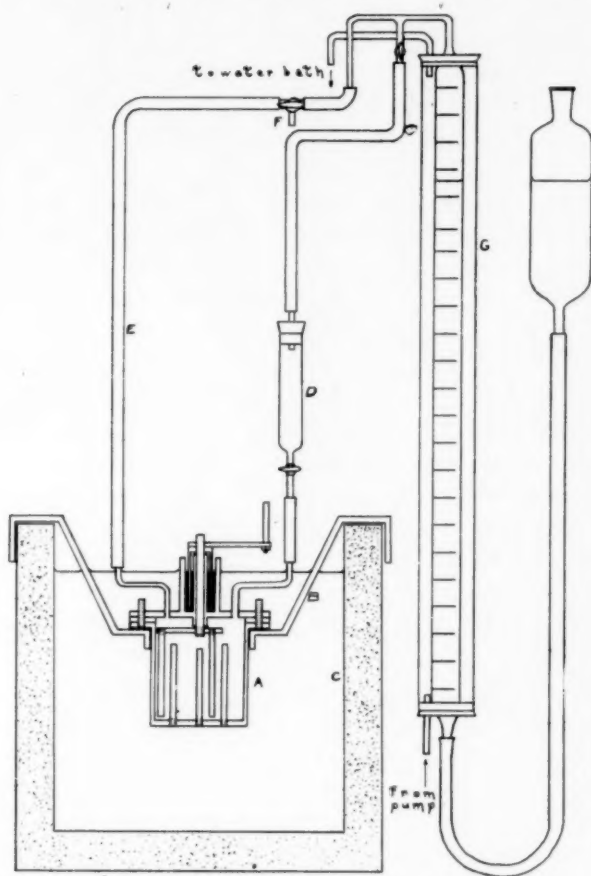


Fig. 1. Apparatus for Rate of Reaction in Dough.

be removed for cleaning purposes. The mixer is supported by a bracket *B* in a water thermostat *C* controlled to  $27^{\circ}\text{C.} \pm 0.5^{\circ}$  in the work presented here. Extending through the lid of the mixer is a copper tube attached by means of pressure tubing to a dropping funnel *D* into which liquid can be introduced and later run into the mixer containing the dry ingredients. The upper end of the dropping funnel has glass and pressure tubing connections with one inlet of the Chittick apparatus *G*. A second such tube connection *E* to the other inlet was found necessary to allow the free flow of liquid into the mixer, thus giving a completely closed system. The leveling liquid of the Chittick apparatus is adjusted by means of stopcock *F* which is closed during a

determination. Temperature control of the Chittick volume measuring tube is maintained by a glass tubing water jacket through which water from the thermostat is circulated by a motor driven water pump. A bypass between the pump and the water jacket not only reduces the pressure in the jacket, but circulates the water in the thermostat. For rate of reaction determinations in biscuit doughs, an aliquot of the dry mix representing 0.75 gram soda was placed in the mixer, the lid clamped down and after attaching to the tubing, a pre-determined quantity of water was run in. Mixing was started as the last of the water entered, time was noted and readings were taken of the volume of the system at time intervals of 1/2, 1, 2 ..., 9, 10, 15 minutes. Doughnut doughs were treated in the same manner except that the mixing time was one minute and at a faster rate of mixing. The experience of the operator is responsible to some extent for reproducibility of results, but variation is negligible when all determinations are made in one day.

A correction was found to be necessary using a control dough, both of the biscuit and doughnut types, in which no leavening ingredients were included. This was due to the fact that when dough ingredients are mixed with water, heat is evolved which, in this case, would cause expansion of occluded air and thus cause an expansion of the dough. Volume measurements are, therefore, presented in corrected form by subtracting values obtained with the control dough from the observations made on biscuit or doughnut doughs.

Replacement of the mixer with a carbon dioxide flask with appropriate support and connections allowed a determination of the rate of reaction of leavening ingredients in water solution. For this purpose baking powders were made up to contain 28% soda and an aliquot, representing 0.75% soda, was acted upon by 10 cc. of water in the flask. A slight rotary motion was given the flask during the first minute and volume readings were taken as in the instance of the doughs.

In the earlier experiments it was found that a variation in atmospheric pressure from day to day affected the duplication of results. Because of this, all volume measurements were taken on dough mixes or baking powders at 27° C., atmospheric pressure was recorded and later calculations were made to a common basis which in this work was 760 mm. Hg. and 0° C. Aliquots representing 0.75 gram bicarbonate of soda were selected due to the fact that the volume of carbon dioxide which should be liberated from sodium bicarbonate on reaction with an acid should occupy 199.976 cc. at 760 mm. Hg and 0° C. Thus, the volume measurements are convertible directly to per cent of the total carbon dioxide available by dividing the calculated and corrected volume



by a factor of two. Since all determinations were made at 27° C. a slight error resulting from the vapor pressure of water over the dough or baking powder solution is possible, but is negligible and has not been corrected for in the data. A second error, also negligible, is that of resistance of a dough to swelling when carbon dioxide is liberated within it. Whether the carbon dioxide is held in solution, is adsorbed by the proteins of the dough, or is held under pressure, the fact remains that it has not escaped from the dough and is there available for expansion of the product upon the application of heat in the oven.

#### RESULTS OF EXPERIMENTS MEASURING RATE OF REACTION

In Table I are presented data typical of that obtained from these experiments for the reaction of sodium acid pyrophosphate with sodium

TABLE I

VOLUME IN CC. OF CARBON DIOXIDE EVOLVED FROM SODIUM ACID PYROPHOSPHATE AND SODA<sup>1</sup>

Time <sup>1</sup>	Water Solution	Biscuit Doughs			Doughnut Doughs		
	A	B	C	D	B	C	D
0	0	0	—	—	0	—	—
1/2	84.3	27.0	—	—	25.6	—	—
1	111.3	42.1	—	—	36.0	—	—
2	120.4	58.1	14.2	43.9	40.5	4.5	36.0
3	122.9	64.9	18.0	46.9	42.9	6.0	36.9
4	124.2	69.9	21.8	48.1	44.7	6.7	38.0
5	124.9	73.8	25.6	48.2	46.0	7.5	38.5
6	125.6	77.5	28.4	49.1	47.5	8.2	39.3
7	125.9	81.0	30.3	48.7	48.1	9.0	40.1
8	126.5	84.3	33.2	51.1	50.5	9.0	41.5
9	127.2	86.6	35.1	51.5	51.9	9.7	41.2
10	127.6	88.8	37.0	51.8	52.0	10.5	41.5
15	129.4	98.2	43.6	54.6	56.7	12.7	43.0

<sup>1</sup> A—Reaction in water.  
B—Reaction in dough.

C—Dough volume.  
D—Volume CO<sub>2</sub> lost.

bicarbonate in water solution as column A and in doughs as column B for both types of doughs. In Figures 2 and 3 are presented curves from the data obtained in the instance of the four baking acids. Curve A represents in each case the rate of reaction in water solution and curve B the rate in doughs. As may be seen, the effect of dough ingredients upon the reaction is very pronounced particularly in the instance of the sodium acid pyrophosphate baking acid. The rate of reaction in water solution would indicate that this acid is a relatively fast acting acid whereas in a dough which more nearly represents actual baking conditions, sodium acid pyrophosphate should be classed as a slow acting baking acid.

The volume of gas measured in the rate of reaction apparatus represents the total gas liberated from doughs as well as dough expansion due to entrapped  $\text{CO}_2$ . In order to determine the actual  $\text{CO}_2$  lost from the dough during mixing, it was necessary to obtain information

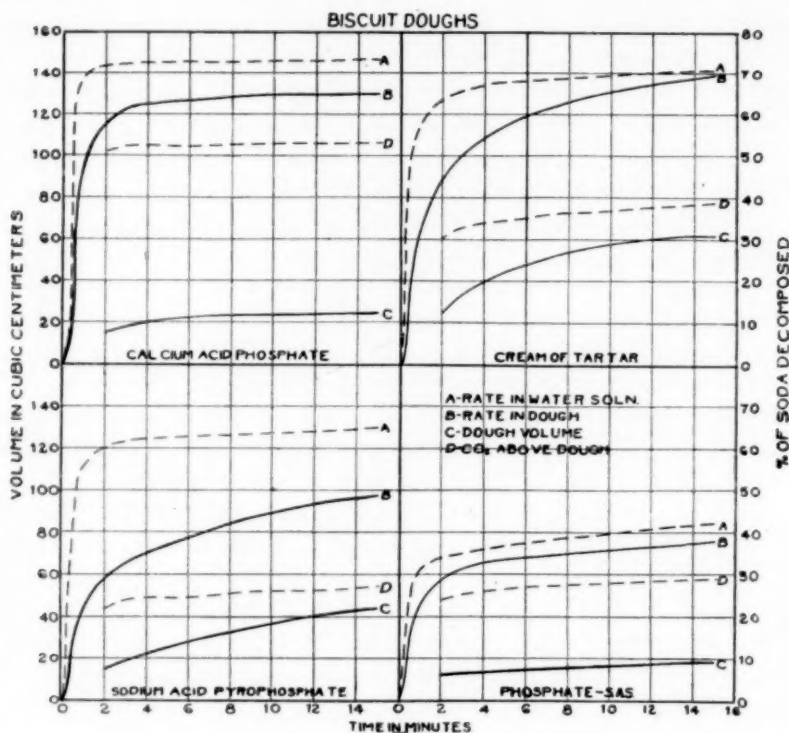


Fig. 2. Carbon Dioxide Evolved or Lost from Water Solution or Biscuit Doughs.

upon the actual expansion of the dough. This was accomplished by mechanically mixing in a Hobart mixer dough batches several times larger than those used in the rate apparatus and placing these in well oiled liter cylinders. The times and severity of mixing and handling were pre-determined to approximate that in the rate mixer. Readings of volume were started two minutes after introduction of liquid to the dry ingredients and were observed each minute as in the instance of rate of reaction experiments. Control doughs containing no leavening were run with both the biscuit and doughnut doughs, and correction was made for purposes outlined above. All doughs were mixed to obtain a temperature of  $27^{\circ}\text{C.} \pm 0.5^{\circ}$  in the final dough. The volumes were then calculated back so as to be equivalent to those of the small batch in the rate of reaction mixer and calculated to  $0^{\circ}\text{C.}$  and 760 mm. Hg.

In Table I, column *C*, are presented data typical of the results obtained by this method which results are presented in Figures 2 and 3 as the *C* curves for each of the baking acids. The slope of the curves indicates a continued reaction over the 2—15 minute period and represents the so-called "bench action." This gas is not lost, but is available for leavening after the dough is placed in the oven. Furthermore, this "bench action" within reasonable limits is desirable and can be compared with the similar action of yeast leavened doughs.

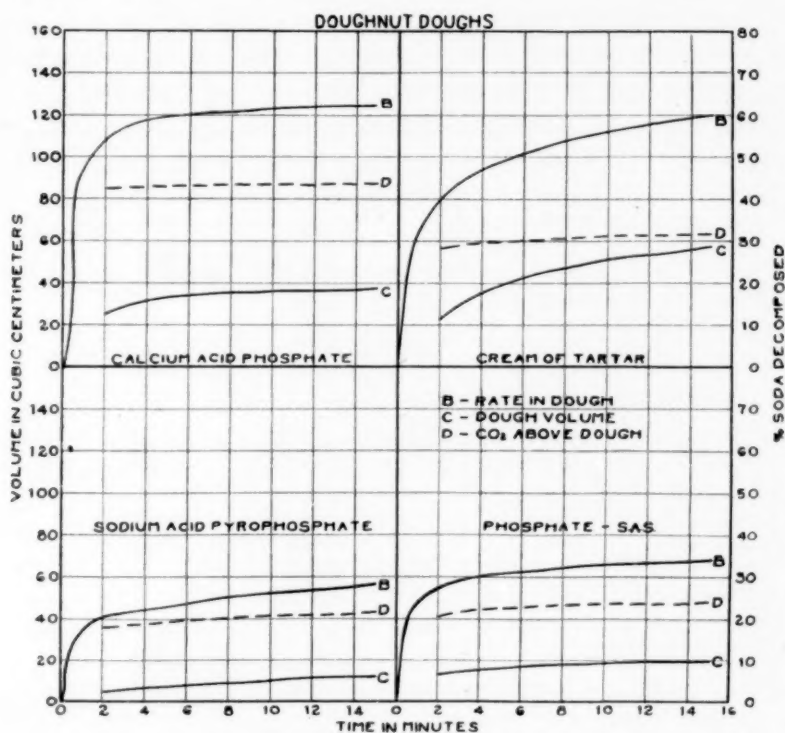


Fig. 3. Carbon Dioxide Evolved or Lost from Doughnut Doughs.

It may be pointed out that the continued reaction of the baking acids with soda is similar in the two types of doughs except in the case of sodium acid pyrophosphate where the reaction is decidedly retarded by the ingredients of the doughnut dough. The initial rise of the curves indicates that the majority of the gas formed during the doughing-up process is lost during the mixing period.

Typical data showing the loss of CO<sub>2</sub> from the dough are presented in Table I, column *D*, and are shown as the *D* curves in Figures 2 and 3 for each of the baking acids.

From these curves it is apparent that a fast acting baking acid

causes a large loss of carbon dioxide from the dough during the mixing process whereas a slow acting acid results in a small loss with a greater quantity of available gas being left for baking. Of the gas generated during mixing only 20% to 30% is retained by the doughs. This, plus the gas in the undecomposed soda, is available for leavening in the oven. It may also be noted from the slope of these curves that no appreciable loss of gas occurs after mixing.

### Results of Baking Tests

Experimental methods and data as here presented can be of value only if they are capable of interpretation as affecting baked products. Two points can be considered for comparison with the data:

1. Baking acids effect a variable loss of leavening gas during the mixing which is reflected in the volume of the baked product.
2. The continuation of reaction during standing of doughs is capable of experimental proof.

Two series of baking tests were selected for this purpose, one using biscuit doughs and the second cake batters.

Two biscuit doughs were made up to contain each of the four baking acids. One dough was mixed, rolled, cut and baked within 2-3 minutes. The second was allowed to stand 15 minutes after mixing, at which time it was rolled, cut and baked. The specific volume of each set of biscuits was then determined by dividing the volume of the biscuits by their weight.

In Table II are presented data taken from that used in the construction of the *D* curves and subtracted from 100% to give the per cent of the total carbon dioxide available in the doughs at the 2 min. period. The per cent of the available carbon dioxide lost over the 2-15 minute period is also given. The specific volume of biscuits baked immediately is given in the table together with the per cent of loss of specific volume after 15 minutes standing of the dough.

TABLE II  
THE PER CENT OF CARBON DIOXIDE LOST FROM BISCUIT DOUGHS COMPARED WITH  
THE REDUCTION IN SPECIFIC VOLUME OF BISCUITS

Acid ingredient	CO <sub>2</sub> available in dough at 2 min.	CO <sub>2</sub> lost after 15 min.	Specific volume of biscuits baked immediately	Reduced specific volume due to stand- ing
	P.ct.	Pct. of available		P.ct. loss
Phosphate	49	4.0	2.24	8.5
S.A.P.P.	78	6.5	2.75	9.0
Tartrate	70	13.0	2.65	11.0
Phosphate—S.A.S. <sup>1</sup>	76	6.5	2.41	3.0

<sup>1</sup> Exception due to effect of acid on colloidal properties of dough.

In general, these baking trials support the conclusion that the loss of leavening gas during the mixing of a dough is a real loss and that the specific volume of baked product is proportional to the entrapped gas and unreacted soda remaining in the dough. Allowing the dough to stand results in continued reaction of the leavening ingredients which, when their product of reaction, carbon dioxide, is removed by manipulation of the dough, causes a reduction in volume.

The methods here employed are capable of extension to the baking of cakes, although extensive investigations have not as yet been undertaken in this laboratory. For illustration, cake batters were made up using a standard formula and including each of the baking acids. A control batter was also mixed in which no leavening agent was included. The carbon dioxide available for baking was determined upon an aliquot of each batter 30 minutes after mixing by means of the Chittick apparatus, and is given in Table III in terms of per cent of total carbon dioxide after correction was made with the control batter figure. The third column represents the increase in specific volume over that of the control cakes. As may be seen from the table, there is a remarkable agreement between the per cent of carbon dioxide available for baking and the volume of the baked product.

TABLE III  
CARBON DIOXIDE CONTENT OF CAKE BATTERS AND THE SPECIFIC VOLUME OF CAKES

Acid ingredient	Per cent CO <sub>2</sub> in batter by analysis	Specific volume of cakes	Specific volume increase over control (1.72)
Phosphate	23.0	2.73	1.01
S.A.P.P.	54.0	3.14	1.42
Tartrate	41.5	2.90	1.18
Phosphate—S.A.S. <sup>1</sup>	52.5	2.89	1.17

<sup>1</sup> Exception due to effect of acid on colloidal properties of dough.

Although the doughnut doughs as employed in rate of reaction determinations were stiffer mixes, a qualitative agreement is observed to exist between the quantity of carbon dioxide retained in those doughs and the quantity of carbon dioxide in cake batters as determined by analysis.

### Discussion

From the foregoing, it will be seen that the major problem connected with determinations of rates of reaction is capable of being solved in dough batches and that a correlation exists between carbon dioxide left in doughs after mixing and the volume of the baked product. No high degree of accuracy is claimed for the method presented here; however, it is capable of interpretation with continued use and

furnishes a means of estimating small differences in rates of reaction of baking acids with baking soda which could not be accomplished by means of water solution tests. A variable of importance which has received little attention is that of the effect of residual salts of baking acid reactions as influencing not only the rate of reaction but also the colloidal properties of the doughs or batters, and finally, the baking results.

That each type of baking acid has its particular application is demonstrated by the wide use of baking powders in the home, restaurant and bake-shop and by the use of baking creams and soda in the bake-shop. The requirements of the type of mix is a determining factor in the use of the various acids. Thus, in the home, prepared biscuit and pancake flours containing calcium acid phosphate are in favor and fulfill the requirements calling for a fast acting acid, since these products are baked immediately and are for immediate consumption. The use of a slower acting acid is a necessity in commercial bake-shops to fit in with their production schedule.

It is believed the method outlined here is of value in demonstrating the characteristics of different types of baking acids or leavening materials and that this information can be applied to many baking problems. In effect, different types of doughs require different types of leavening materials and modifications in the manner of handling the doughs can affect the results obtained. It follows that any user of leavening materials has the choice of selecting the baking acid to fit his particular needs; or, he may adapt his formula and baking procedure to fit the characteristics of the particular type of baking acid which he is using.

### Summary

1. An apparatus has been described for determining the rate of reaction of chemical leavening agents, known commercially as baking powders, under actual conditions of use in the bake-shop or home.
2. A method has been presented to obtain the volume of dough expansion in the rate apparatus, the carbon dioxide lost from the dough, and by difference the per cent of total available carbon dioxide remaining in the dough for oven expansion.
3. Baking trials have been made which are in general agreement with the results obtained in rate of reaction and dough volume experiments.
4. The major part (70% to 80%), of the carbon dioxide generated from baking powders during the mixing of doughs, is lost. The amount so generated varies with the acid used. The remainder of the gas is retained in the dough together with unreacted soda for expansion and reaction at oven heat.



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## A REVIEW OF PATENTS AND THE APPLICATION THEREOF INVOLVING IRRADIATED CEREAL PRODUCTS

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(Read at the Convention, May, 1931)

Patent grants dealing with irradiation of foods by ultra violet light involve equipment as well as process patents.

Assuming that your interest in process patents is greater than that in those affecting the equipment, a special effort has been made to analyze and abstract the process patents instead of attempting to give a digest of all the patents pertaining to irradiation with ultra violet light.

The patent that has attracted the greatest public interest is that entitled "Anti-rachitic Product and Process" granted to Harry Steenbock, Madison, Wis., by the British Government on June 30, 1924; by the United States Government on August 14, 1928, and by the Canadian Government, July 9, 1929.

This patent provides for imparting anti-rachitic properties to dietary organic substances such as carbohydrate foods, fats, oils, protein foods or composite foods. Proposal is made that these foods be subjected to the ultra violet rays produced by a quartz mercury vapor lamp for a period sufficient to effect anti-rachitic activation but so limited as to avoid subsequent deterioration of the anti-rachitic substances or of the palatability of the foods so treated. Specific application of this process designates treatment of: (1) oils and fats containing unsaponifiable lipoids; (2) liquid glycerides naturally deficient in anti-rachitic properties; (3) olive oil, and (4) food substances naturally deficient in anti-rachitic substances.

Although the application for patent grants were filed 38 months apart, on the same date, August 14, 1928, patent rights were granted

August J. Pacini, Chicago, Illinois, for the production of anti-rachitic substances and their products.

This grant merely provides for the preparation of materials containing growth-producing substances in a manner so that at least some of these growth-producing substances will be replaced by anti-rachitic products, the method of treatment of growth-producing substances with rays of light having a wave length greater than about 3022 Ångström units.

This grant to Pacini is amplified by another one dated June 3, 1930, in which his application of ultra violet radiation treatments includes wave lengths beginning at about 4000 Ångström units and extending throughout the near, middle and far zones of spectrum rays, including those of the so-called "border-line" rays where X-rays and ultra violet rays become inseparable, which is to say that all rays having wave lengths from 4000 to 500 Ångström units (0.4 to 0.05 microns) are included.

Further definition is given to Pacini's claims by the patent granted July 22, 1930, in which he provides for the formation of vitamin D from sterol-containing substances by radiation with wave lengths not shorter than 3022 Ångström units (0.3022 microns) by making use of such catalyzing agents as chlorine, bromine, iron salts, uranium salts, titanium salts, ozone, certain unnamed organic dye-stuffs and other unnamed substances.

Although four of the five claims of this patent grant involve the use of a photo-catalyst, the fifth claim provides for preparing sterols so that they become anti-rachitic through treatment with rays longer than about 3022 Ångström units (0.3022 microns). In text of the patent specific dissertations are given concerning the treatment of growth-producing substances with radiations of light having wave lengths beginning at 3022 and extending to 2000 Ångström units from which all shorter wave lengths have been filtered out by ordinary glass; with radiations of light from a Cooper-Hewitt work lamp operating on 110-volt direct current for a period of two hours; with radiations of light from a quartz mercury vapor lamp passed through an ordinary window glass to the material treated; with infra-red radiations longer than 9000 Ångström units, not glass filtered, for a long exposure period; with radiations of heat having wave lengths of 3300 Ångström units (shorter wave lengths being filtered out); with radiations of same intensity having wave lengths that lie in region greater than 12000 Ångström units; with radiations of monochromatic light in the region absorbed by the material; with radiations from sunlight for long period of time; with radiations of cathode rays of ordinary Lenard or X-ray tubes and with radiations from radio-active substances such

as thorium, radium or uranium for the production of anti-rachitic substances. Specific reference is made to extracts of growing organisms such as certain fungi and bacteria, sarcomatous tissue, and ergot of rye; to ergosterol, ordinary cholesterol, corn, linseed, peanut and other seed oils; to cereal products, including rolled oats, cornflakes, bran flakes and co-related goods; to cocoa butter alone or in chocolate and similar products, and to such amino-acid substances as tyrosine.

Prior to the last two patents granted Pacini on June 3 and July 22, 1930, Jacques W. D. Chesney, Chicago, Illinois, on March 5, 1929, was granted a patent in which he proposes a method of utilizing the ultra violet rays of the sun for the production of vitamin D in substances susceptible to such activation. Chesney proposes separation of the solar ultra violet rays having wave lengths within the range of substantially 2900 to 3700 Ångström units and intensification by passing them through lenses for concentration upon material to be treated.

Subsequent to this grant, yet prior to the last two Pacini patents, Chesney, on August 6, 1929, was awarded a patent for the protection of vitamin C during process of sterilization and activation of liquid and solid foods with ultra violet rays. By this process the material is acidified with less than 1% of a mild organic acid and exposed to ultra violet rays of not less than 2000 Ångström units. In case of milk and ice cream mixes, oxygen of air is excluded to prevent formation of ozone and impartation of objectionable odor and flavor. In all cases infra-red rays and ultra violet rays shorter than 2000 Ångström units are filtered out.

Subsequent to the other patents mentioned and also prior to the last two patents granted Pacini, Joseph Perino, Stettin, Germany, was granted a U. S. A. patent on April 15, 1930, in which he describes a process of manufacturing a food product by exposing vegetable, alimentary material containing soluble phosphates to ultra violet rays and heating to a temperature not exceeding 60° C. in an atmosphere of reduced pressure and low in oxygen content—more specifically in an atmosphere of CO<sub>2</sub>.

On July 10, 1928, approximately one month before Steenbock was granted his patent, George Sperti and co-workers at University of Cincinnati, Ohio, were issued a patent covering the sterilization and activation of food by selective irradiation by ultra violet light. Although issued before the Steenbock patent, application for this grant was not filed until approximately two and one-half years after Steenbock had filed his claims.

In this patent, which was assigned to a trustee representing the University of Cincinnati and which gave rise to the announcement in

January, 1931, regarding the formation of General Development Laboratories, Inc., by the University of Cincinnati, and General Foods Corporation, Sperti and his co-workers select quantitatively the specific range of ultra violet rays that tests show to be specific in action to give the desired results.

Sperti and his associates state that their work showed bacteria to be killed by light rays with wave lengths beginning at approximately 2750 Ångström units; that enzymes were rendered inactive by light rays having wave lengths of 2420 Ångström units and shorter; that radiations of wave lengths greater than 2750 Ångström units did not kill bacteria; that by utilizing light rays with wave lengths greater than 2400 and not greater than 2750 Ångström units the bacteria were killed without inactivating the enzymes and that a filter screen filled with a 5% solution of lead acetate in pure water gave the desired effect.

They also set forth that bacteria in a substance containing a diastatic enzyme were killed without affecting the enzymes; that enzymes were prevented from converting sugars into alcohols during fermentation by gas-producing bacteria; that protozoa were destroyed in solutions without changing the desirable materials in the solution and that a suspension of yeast in a thin film can be subjected to the filtered light from the quartz lamp for a prolonged period of treatment without deleterious effect or by repeated instead of prolonged exposures.

Becoming more specific they formulated the procedure for activating portions of substances without activating undesirable portions by determining the critical action for each substance. In treating cod liver oil to activate the vitamin D a specific and critical band of light radiations would give the desired result without danger of inactivating the vitamin because of over-exposure. Claim was made that it will be possible for the investigator to use the various filters now available or made by himself in determining what will be the critical wave band for any substance with which he is endeavoring to work and for the elements that make it up.

Although some commercial irradiation of foods is being done on an unadvertised and unofficial basis, relatively speaking, the Wisconsin Alumni Research Foundation, assignee of the Steenbock patent, is the only holder of these irradiation patents that has reduced the art to practice. Directly from the Wisconsin Alumni Research Foundation the Quaker Oats Company has obtained license to irradiate at least two of its cereal products; the Fleischmann Company, its foil wrapped yeast and dry yeast product as well as its ergosterol, and four U. S. A. and three Canadian concerns producing viosterol for medicinal or drug purposes.

Indirectly, through the National Foods, Ltd., Toronto, Canada, composed of Wisconsin Alumni Research Foundation and the personnel of the Paediatric Research Foundation of University of Toronto, Canada, the General Baking Company has obtained exclusive right for use of irradiated products in bread making.

In all cases, regarding the issuance of licenses for operating under the Steenbock patent, the Wisconsin Alumni Research Foundation stipulates that the admission of irradiation products into foods shall be of sufficient quantity to leave no question as to the presence of vitamin D, although the potency of the food product must be kept appreciably below that which would be required for a therapeutic product such as cod liver oil or viosterol in oil.

Although the General Development Laboratories, Inc., has not as yet made commercial application of the Sperti process, plans and arrangements are progressing to that end and public announcement of perfected plans need not come as a surprise.

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#### NEW PUBLICATION

Commencing with the August 6th issue, the journal "Die Mühle" has issued a monthly supplement entitled "Das Mühlenlaboratorium." Das Mühlenlaboratorium is devoted entirely to purely scientific articles on cereal chemistry. In addition, it contains a worthwhile abstract section. Das Mühlenlaboratorium may be obtained by addressing Hans Kunis, Salomonstr. 8, Leipzig, C 1/23, Germany.

## EMPLOYMENT INFORMATION

*The American Association of Cereal Chemists in convention at St. Louis, Mo., in June, 1925, voted to establish an employment service. A committee was appointed to administer this service of which Mr. C. B. Morison, 1135 Fullerton Avenue, Chicago, Ill., is chairman. Members of the association are extended the privilege of utilizing the services of this committee, and of publishing without charge suitable advertisements for Situations Wanted. CEREAL CHEMISTRY will publish these advertisements after they have been submitted to and approved by the Employment Committee. Correspondence and copies of advertisements should be sent to the Chairman of the Committee.*

*Non-members of the association may advertise in this journal for positions upon payment of one dollar per insertion of four lines plus twenty-five cents for each additional line.*

*Prospective employers may address the Chairman of the Employment Committee for such information as has been furnished the committee concerning the qualifications of the chemists who are seeking employment.*

*CEREAL CHEMISTRY will publish without charge advertisements of "Situations Open," and invites employers of chemists to utilize this service.*

Single issues of THE JOURNAL of the AMERICAN ASSOCIATION OF CEREAL CHEMISTS wanted.

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